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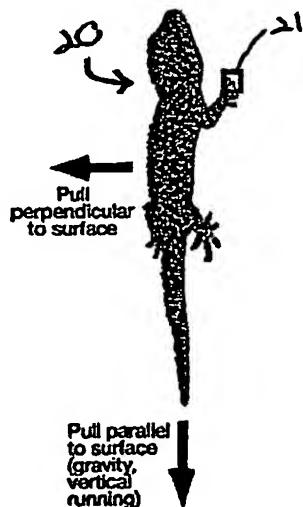
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(54) Title: ADHESIVE MICROSTRUCTURE AND METHOD OF FORMING SAME



(57) Abstract: A method of forming an adhesive force includes removing a seta from a living specimen, attaching the seta to a substrate, and applying the seta to a surface so as to establish an adhesive force between the substrate and the surface. The seta is applied to the surface with a force perpendicular to the surface. The seta is then pulled with a force parallel to the surface so as to preload the adhesive force of the seta.

WO 01/49776 A2

## **ADHESIVE MICROSTRUCTURE AND METHOD OF FORMING SAME**

This application claims priority to the provisional patent application entitled, "Non-Interlocking Dry Adhesive Microstructure and Method of Forming Same," Serial Number 60/172,731, filed December 20, 1999.

This invention was made with Government support under Grant (Contract) No. N00014-98-C0183 awarded by DARPA through a subcontract from I.S. Robotics. The Government has certain rights to this invention.

### **BRIEF DESCRIPTION OF THE INVENTION**

This invention relates generally to the fabrication and utilization of micron-scale structures. More particularly, this invention relates to a non-interlocking adhesive microstructure.

### **BACKGROUND OF THE INVENTION**

There is an ongoing need for improved adhesives. Improved adhesives have applications ranging from everyday aspects of life (e.g., tape, fasteners, and toys) to high technology (e.g., removal of microscopic particles from semiconductor wafers, transporting fiber optic devices, and assembly of sub-mm mechanisms, particularly those including micro-fabricated components, or components that cannot tolerate grippers, adhesives, or vacuum manipulators).

Adhesive mechanisms in nature have been studied, but have not been fully understood or exploited. For example, Geckos are exceptional in their ability to rapidly climb up smooth vertical surfaces. The mechanism of adhesion used in Geckos, *Anolis* lizards, some skinks, and some insects, has been debated for nearly a 5 century.

While some prior work has identified the morphology of seta used by Geckos and other insects, this prior work does not identify how the seta operates. In addition, this prior work fails to identify how to use a seta to perform useful work.

It would be highly desirable to identify and exploit the adhesive force 10 mechanism utilized by Geckos and other insects. Such information could result in the utilization of new adhesive microstructures and the fabrication of such structures.

### SUMMARY OF THE INVENTION

The invention includes a method of forming an adhesive force. The method 15 includes the steps of removing a seta from a living specimen, attaching the seta to a substrate, and applying the seta to a surface so as to establish an adhesive force between the substrate and the surface.

The invention also includes a method of establishing a adhesive 20 microstructure. The method includes the step of applying a seta to a surface with a force perpendicular to the surface. The seta is then pulled with a force parallel to the surface so as to preload an adhesive force of the seta.

The invention also includes a method of fabricating an adhesive 25 microstructure. The fabrication technique includes fabricating an array of shafts and then forming spatulae on the array of shafts.

The invention also includes a fabricated microstructure with a shaft having a 30 length of less than 500 microns. The shaft has a diameter of between 0.01 and 0.1 times the length of the shaft. An array of spatulae is formed at an end of the shaft. The array of spatulae has a width of less than 10 microns. Individual spatula of the spatulae include a terminal end with an extended surface, such as a paddle, a curved segment of a sphere, or a flattened segment of a sphere.

**BRIEF DESCRIPTION OF THE DRAWINGS**

For a better understanding of the invention, reference should be made to the following detailed description taken in conjunction with the accompanying drawings, in which:

5 FIGURE 1A illustrates preloading operations performed in accordance with an embodiment of the invention.

FIGURE 1B illustrates rows of setae used in accordance with the invention.

FIGURE 1C illustrates a single seta used in accordance with the invention.

10 FIGURE 1D is an enlarged view of a single seta used in accordance with an embodiment of the invention.

FIGURE 1E is an enlarged view of a single extended surface spatula on a spatula stalk, in accordance with an embodiment of the invention.

FIGURE 1F is an enlarged view of a single extended surface spatula on a spatula stalk, in accordance with an embodiment of the invention.

15 FIGURE 1G illustrates an array of spatulae formed at the end of a shaft to form seta used in accordance with the invention.

FIGURE 1H illustrates a system to measure adhesive force achieved in accordance with the invention.

20 FIGURE 1I is another illustration of a system to measure adhesive force achieved in accordance with the invention.

FIGURES 2A-2B illustrate different forces, as a function of time, associated with the loading and adhesion operation of a structure of the invention.

FIGURE 3 illustrates perpendicular preload force associated with an embodiment of the invention.

25 FIGURE 4 illustrates perpendicular force during detachment of a structure utilized in accordance with the invention.

FIGURES 5A-5C illustrate the fabrication of an array of setae with spatula structures in accordance with an oxide/nitride fabrication process utilized in accordance with an embodiment of the invention.

FIGURES 6A-6B illustrate the fabrication of an array of setae in accordance with an excitation source process utilized in accordance with an embodiment of the invention.

5 FIGURE 7 illustrates the fabrication of an array of setae with spatula structures in accordance with a stalk and seeding process utilized in accordance with an embodiment of the invention.

FIGURE 8 illustrates the fabrication of a single spatula using a micro-pipette.

FIGURE 9 illustrates an embossing tool used to form a spatulae mold for use in accordance with an embodiment of the invention.

10 FIGURES 10A-10D illustrate lithographically induced self-construction of spatulae in accordance with an embodiment of the invention.

FIGURES 11A-11B illustrate a roller nano-imprinting technique that may be used to form spatulae in accordance with an embodiment of the invention.

15 FIGURE 12 illustrates a two-layer photoresist fabrication technique that may be used in accordance with an embodiment of the invention.

FIGURE 13 illustrates a setae-based manipulator that may be used in accordance with an embodiment of the invention.

Like reference numerals refer to corresponding parts throughout the drawings.

## 20 DETAILED DESCRIPTION OF THE INVENTION

The invention is directed toward the use of micron scale structures to achieve adhesion. In particular, the invention uses a seta structure. The seta structure has a shaft. Positioned at the end of the shaft is a spatula or an array of spatulae. Adhesion is produced as the spatula or array of spatulae produce intimate contact with a surface.

25 In general, the shaft is between 1 and 500 microns long, preferably approximately 10 to 100 microns long. The diameter of the shaft is preferably between 0.01 and 0.1 times the length of the shaft, preferably approximately 0.05 times the length of the shaft.

30 The terminal end of the shaft has at least one spatula. Preferably, the terminal end of the shaft has between 1 and 1000 spatulae. The array of spatulae is preferably less than 10 microns wide, preferably approximately 1 micron wide. Preferably, each

spatula of the array of spatulae has an extended surface at its terminal end. The extended surface may be in the form of a paddle or a curved segment of a sphere, as shown below.

The structure of the invention is modeled from structures found in nature, such 5 as the seta found on the foot of a Tokay gecko (*Gekko gecko*). Many species of gecko (e.g., clade Gekkonoidea), species of *Anolis*, and several skink species have adhesive setae that may also be used in accordance with the invention. In addition, beetles and kissing-bugs have setae that may be used in accordance with the invention. The invention is implemented with natural or fabricated setae, as discussed below.

10 Examples of seta structures found in nature follow. The seta of a Tokay Gecko has a stalk (shaft) diameter of  $5\mu$ , a stalk height of  $110\mu$ , a tip (spatulae) length of  $0.2\mu$ , a tip width of  $0.2\mu$ , and between 100-1000 tips, where the total tip area per stalk is 2 to  $20\mu^2$ . *Anolis cuvieri* has a stalk diameter of  $0.5\mu$ , a stalk height of  $22\mu$ , a tip length of  $0.6\mu$ , a tip width of  $0.7\mu$ , and between 100-1000 tips, where the 15 total tip area per stalk is 2 to  $20\mu^2$ . *Prasinohaema virens* (skink) has a stalk diameter of  $2\mu$ , a stalk height of  $26\mu$ , a tip length of  $6\mu$ , a tip width of  $7\mu$ , and between 100-1000 tips, where the total tip area per stalk is approximately  $20\mu^2$ .

By way of example, Figure 1A illustrates a Tokay gecko 20 with terminal limbs 21 that have naturally occurring setae. The live gecko 20 is restrained. The 20 cuticular layer of a portion of a terminal limb (e.g., a toe) 21 is removed. This operation, analogous to cutting hair, allows the gecko to harmlessly regenerate its setae. It has been demonstrated that hundreds or thousands of setae can be easily harvested without sacrificing the living being from which the setae are removed.

After removal, the cuticular surface is scraped to break off individual seta, 25 preferably at the base of the shaft of the seta. Figure 1B illustrates rows 22 of setae associated with the gecko 20. Figure 1C illustrates the shaft 24 of a single seta 26. The figure also illustrates the spataulae 28 positioned at the end of the shaft 24.

Figure 1D is an enlarged view of a single seta 26. The figure illustrates that the shaft 24 is roughly perpendicular to the spatulae 28.

30 Figure 1E is an enlarged view of a single spatula 29 on a spatula stalk 30. The spatula stalk 30 may be the shaft 24 or a separate tendril extending from the shaft 24.

Preferably, each spatula 29 has an extended surface. In Figure 1E, the extended surface is in the form of a paddle structure. In Figure 1F, the extended surface is in the form of a sphere. Figure 1G is an enlarged view of an array of spatulae 28.

The realization of large adhesive forces by the setae is contingent upon preload operations performed in accordance with the invention. Detachment of the setae occurs at a characteristic angle, as specified in accordance with the invention. Figure 1H illustrates a substrate (i.e., a sensor 32) that was used to characterize these forces. Figure 1I illustrates the characteristic angle ( $\alpha$ ) for detaching setae of the invention. The characteristic angle ( $\alpha$ ) is formed between the seta 26 and a surface 40 to which the seta is attached.

The inventors have identified that the adhesive force of a seta depends upon its three-dimensional orientation (spatulae pointing toward or away from the surface) and the extent to which the seta is preloaded (pushed into and pulled along the surface) during initial contact. Contacting the surface with the seta in a direction other than with spatulae projecting toward the surface resulted in forces less than 0.3  $\mu$ N when the seta was pulled away perpendicular to the surface. A pull parallel to the surface showed that the force produced by the inactive, non-spatular region increased with normal or perpendicular force, typical of a material with a coefficient of friction equal to 0.2. By contrast, when the active spatular region was projecting toward the surface, force increased by 20 to 60-fold. The force resulting from pulling the seta parallel to the surface during attachment increased when setae were first pushed toward the surface, providing a perpendicular preloading force. This initial perpendicular force need not be maintained during the subsequent pull. Setal force parallel to the surface increased linearly with the perpendicular preloading force.

Experiments in which seta were pulled away from the surface of a wire demonstrated that perpendicular preloading alone is insufficient to prevent the seta from being dislodged easily. Seta that were first pushed into the surface and then pulled parallel to it developed over ten times the force (13.6  $\mu$ N  $\pm$  2.6 SD; N = 17) upon being pulled away from the surface than those having only a perpendicular preload (0.6  $\mu$ N  $\pm$  0.7 SD; N = 17). The largest parallel forces were observed only following a few microns of sliding. The results of preloading on setal force

production suggest that a small perpendicular preloading force in concert with a rearward displacement or parallel preload may be necessary to "engage" adhesion. Preloading is believed to increase the number of spatulae contacting the surface.

The orientation of the setae is also important in detachment. The force produced when a seta was pulled away from the surface was not significantly differently from the force measured during a pull parallel to the surface if the same perpendicular preload was given. However, it has been identified that setae detached at a similar angle ( $30.6^\circ \pm 1.8$  SD; N = 17) and force when pulled away from the sensor's surface. To check for the presence of a critical angle of detachment, the perpendicular force was held constant, while the setal angle was progressively increased until detachment. Setal angle at detachment changed by only 15% over a range of perpendicular forces. Thus, the invention utilizes a detachment angle of between  $35^\circ$  and  $25^\circ$ , preferably approximately  $30^\circ$ . The detachment angle values are based upon the disclosed seta structure in which the shaft of the seta is roughly perpendicular to the spatular surface, as shown in Figure 1D. Change in the orientation of the setae and perhaps even the geometry of the spatulae may facilitate detachment.

The foot of a Tokay gecko (*Gekko gecko*) holds approximately 5000 setae  $\text{mm}^{-2}$  and can produce 10 N of adhesive force with approximately  $100 \text{ mm}^2$  of pad area. Therefore, each seta should produce an average force of  $20 \mu\text{N}$  and an average stress of  $0.1 \text{ N mm}^{-2}$  ( $\sim 1$  atm). The actual magnitudes are probably greater, since it is unlikely that all setae adhere simultaneously.

The foregoing information is more fully appreciated in connection with specific operations performed in accordance with the invention. An isolated seta, secured by the technique discussed above, was glued to a substrate (e.g., to the end of a #2 insect pin) with epoxy (e.g., 5-MINUTE EPOXY sold by TTWDevcon, Danvers, MA). The pin had a diameter of approximately  $15 \mu\text{m}$ . To prevent the epoxy from creeping up the stalk of the seta, which might change the mechanical property of the specimen, the epoxy is preferably precured for approximately 1 minute before applying it to the specimen. All setae were oriented such that the active surface was

approximately perpendicular to the axis of the pin. All preparations were completed under a compound microscope.

Force production by single, isolated seta during attachment was measured using a micromachined, dual-axis, piezoresistive sensor 32 of the type illustrated in 5 Figure 1H. The following discussion provides information with respect to the sensor 32. U.S. Patent 5,959,200 describes a sensor of the type described herein. The sensor 32 does not form a part of the invention, rather it is merely used to obtain the performance results described below.

The cantilever sensor 32 of Figure 1H was fabricated on a single-crystalline 10 silicon wafer. The cantilever 32 has two independent force sensors, each with one predominant direction of compliance. The perpendicular force sensor consists of a thin triangular probe 50. The parallel force sensor is composed of four long slender ribs 52. A special 45° oblique ion implantation allowed piezoresistive and conductive regions to be implanted on both the parallel and perpendicular surfaces 15 simultaneously. Forces applied to the tip of the sensor were resolved into these two orthogonal directions (parallel and perpendicular), and were measured by the changes in resistance of the piezoresistors. Since this device was originally designed for Atomic Force Microscope data storage applications, each of these cantilever devices had a sharp tip near the vertex of its triangular probe. For the gecko setae adhesion 20 measurement, the back-side of this device was used to provide a smooth surface for setal adhesion.

Each seta 26 was brought in contact with the sensor 32 by applying a small preload perpendicular to the surface to increase contact and induce adhesion. To determine the effect of preload force on submaximal parallel force, preload force was 25 varied when setae were attached to the tip of the sensor, as shown in Figure 3, which is discussed below. To measure maximal parallel force, the base of the triangular probe was used. Using the base increased area of contact, but did not allow for simultaneous measurement of preload forces. Sensor signals were taken while the seta was being pulled parallel to the surface by a piezoelectric manipulator at a rate of 30 ~5  $\mu\text{m sec}^{-1}$ . Sensor signals were amplified and filtered through a 300-Hz low-pass filter, and then digitized at 100 Hz using a 16-bit data acquisition card (LabView™ on

a PC). The collected data (in volts) were converted to deflections of the sensor through calibration constants, and multiplied by the stiffness of the cantilever to obtain force values.

Breaking or detachment force was defined as the maximal force a seta could exert perpendicular, or normal, to a surface immediately before it released. This value was determined for individual seta by measuring the amount it could displace a force gauge made from a 4.7 mm aluminum bonding wire with 25  $\mu\text{m}$  nominal diameter (American Fine Wire Corp., Selma, AL; the wire 40 is shown in Figure 11). To maximize contact area of the active surface of the seta to the wire, a 50  $\mu\text{m} \times 100 \mu\text{m}$  section of the wire tip was flattened. The proximal end of the wire was fixed with epoxy onto a brass stub. The active surface of the seta was pressed against the flattened wire, producing a known perpendicular preload ( $1.6 \pm 0.25 \mu\text{N}$ ; mean $\pm$ SD). The force was measured using two different methods of detachment: (1) the seta was pulled normal to the wire; and (2) the insect pin was displaced  $19.7 \pm 3.45 \mu\text{m}$  along the wire to produce an additional parallel preload on the seta before pulling perpendicular or normal to the wire.

In all trials, detachment force was calculated from the maximum displacement of the wire pulled by the seta. All sequences were recorded with a video camera (e.g., a CCD camera sold by SONY) and digitized to a computer (e.g., an APPLE, MACINTOSH) using a video editing system (e.g., from MEDIA 100 Inc., Marlboro, MA). The initial position of the wire, the angle of the seta with respect to the wire and the position of the wire at the point of separation were recorded and analyzed using image analysis software (e.g., NIH-Image software). The amount of deflection in the force gauge was converted to adhesion force after the force gauge was calibrated against standard weights.

The results of these operations are shown in Figures 2-4. Figure 2A illustrates forces associated with a perpendicular preload and a subsequent parallel pulling performed in accordance with the invention. As shown in Figure 2A, setal adhesive force parallel to the surface increased linearly until the seta began to slide off the edge of the sensor at time  $t_s$ . If the seta was allowed to slide approximately 5  $\mu\text{m}$  along the sensor's surface, a distance imperceptible at the level of the foot, adhesive force

continued to increase, as shown in Figure 2B. The maximum adhesive force of single seta averaged  $194 \mu\text{N} \pm 25 \text{ SD}$  ( $N = 28$ ), nearly 10-fold greater than predicted from whole animal estimates.

The single-seta force measurements suggest that if all setae were  
5 simultaneously and maximally attached, a single foot of a gecko could produce 100 N  
of adhesive force (-10 arm). Stated another way, the foot of a gecko could generate  
maximum observed force (10 N) with only 10% of its setae maximally attached.

The maximum force developed by a given seta could not be predicted from  
molecular interactions or microscopic anatomy alone. Setal force depended on its  
10 three-dimensional orientation (spatulae pointing toward or away from the surface) and  
the extent to which the hair was preloaded (i.e., pushed into and pulled along the  
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perpendicular preloading force, as shown in Figure 3. Experiments in which seta  
were pulled away from a surface (e.g., surface 40, a wire in Figure 1F) demonstrated  
25 that perpendicular preloading alone is insufficient to prevent the seta from being  
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away from the surface than those having only a perpendicular preload ( $0.6 \mu\text{N} \pm 0.7 \text{ SD}$ ;  $N = 17$ ). The largest parallel forces were observed only following a few microns  
30 of sliding, as shown in Figure 2B.

The results of preloading on setal force production support the hypothesis that a small perpendicular preloading force in concert with a rearward displacement or parallel preload may be necessary to “engage” adhesion. Since the tips of the setae are directed rearwards away from the toenail, preloading may increase the number of 5 spatulae contacting the surface.

The orientation of the setae also appears to be important in detachment during locomotion. The force produced when a seta was pulled away from the surface was not significantly different from the force measured during a pull parallel to the surface if the same perpendicular preload was given. However, it was identified that setae 10 detached at a similar angle ( $30.6^\circ \pm 1.8$  SD; N = 17) when pulled away from the wire sensor’s surface. To check for the presence of a critical angle of detachment, perpendicular force was held constant, while the setal angle ( $\alpha$ ; Figure 1F) progressively increased until detachment. Setal angle at detachment changed by only 15% over a range of perpendicular forces, as shown in Figure 4. This observation is 15 consistent with an adhesive model where sliding stops when pulling at greater than the critical setal angle and hence stress can increase at a boundary, causing fracture of the contact. Change in the orientation of the setae and perhaps even the geometry of the spatulae may facilitate detachment.

It has long been known that geckos peel the tips of their toes away from a 20 smooth surface during running. Toe peeling may have two effects. First, it may put an individual seta in an orientation or at a critical angle that aids in its release. Second, toe peeling concentrates the detachment force on only a small subset of all attached setae at any instant. The toe peeling behavior is analogous to the technique used by humans to remove a piece of tape from a surface.

The direct setal force measurements are consistent with the hypothesis that 25 adhesion in geckos is the result of intermolecular forces. The simple models available can only give the most approximate estimates of setal force production. If it is assumed that the tip of a spatula is a curved segment of a sphere (radius,  $R = 2\mu\text{m}$ ) and is separated by a small distance from a large, flat surface where van der Waals 30 forces become significant (atomic gap distance,  $D \approx 0.3$  nm), then setal force =  $AR / 6D^2$ , where A is the material dependent Hamaker constant taken to be  $10^{-19} \text{ J}^{10}$ . This

estimate puts the van der Waals force for a spatula to be about  $0.4 \mu\text{N}$ . Since the number of spatula per seta varies from 100 to 1000, setal force estimates range from 40 to  $400 \mu\text{N}$ .

Earlier experimental support for the van der Waals hypothesis comes from the 5 observation that adhesive force of a whole gecko increases with increasing surface energy of the substrate. In addition, the rejection of alternative mechanisms such as suction, electrostatics, friction, microinterlocking, and wet adhesion, has been attempted. Adhesion experiments carried out in a vacuum and the disclosed measurements of greater than one atmosphere of adhesion pressure strongly suggest 10 that suction is not involved. Experiments using X-ray bombardment eliminates electrostatic attraction as a mechanism necessary for setal adhesion, since the setae can still adhere in ionized air. Microinterlocking could function as a secondary mechanism, but the ability of geckos to adhere to polished glass shows that irregularities on the scale of the spatulae are not necessary for adhesion. The findings 15 herein do not support a friction mechanism because the cantilever's surface is smooth (surface roughness less than or equal to  $2.5 \text{ nm}$ ) and the coefficient of friction of the setal keratin on silicon is low ( $\mu = 0.25$ ; Fig. 3; dashed line). Capillary adhesion or glue are not likely mechanisms, since skin glands are not present on the feet of lizards. The mechanism of adhesion may involve a thin layer of water, or adsorbed water 20 molecules on the seta and/or substrate.

Van der Waals forces are extremely weak at greater than atomic distance gaps, and require intimate contact between the adhesive and the surface. Polymeric adhesives such as tape are soft, and are able to deform sufficiently for intimate contact over a relatively large surface area. The feet of a Tokay gecko (*Gekko gecko*) contain 25 approximately one billion spatulae that appear to provide a sufficiently large surface area in close contact with the substrate for adhesion to be the result of van der Waals forces.

As previously indicated, the invention may be used in connection with setae harvested from a live specimen. Alternately, the techniques of the invention may be 30 used in connection with fabricated setae. Those skilled in the art will recognize a number of techniques that may be used to fabricate setae in accordance with the

invention. For example, the devices may be fabricated through an oxide/nitride process, as shown in Figures 5A-5C.

Initially, a recess is etched in a semiconductor substrate. Figure 5A illustrates a recess 101 formed in a semiconductor substrate 100. Nitride and oxide layers are 5 then deposited on the substrate 100. Figure 5A illustrates a nitride layer 102 and an oxide layer 104. The surface is then patterned and etched, resulting in the structure of Figure 5B.

Afterwards, the underlying substrate 100 is etched, resulting in a well 106, as 10 shown in Figure 5C. At this point, the stress difference between the oxide and nitride layers causes the structure to curl from the plane defined by the substrate 100, thereby forming a shaft structure. The end of the shaft may then be roughened to form spatulae. For example, the spatulae may be formed by wet etching, radiation, plasma roughening, electro-chemical etching, and the like. Alternately, a separate spatulae may be affixed to the shaft. Techniques for fabricating spatulae are discussed below.

15 Another technique that may be utilized in accordance with the invention exploits an excitation source. As shown in Figure 6A, a sensitive material 122 is formed on a substrate 120. An excitation source 124 is used to apply excitation energy to the sensitive material 122. The deep-penetrating excitation alters the volume along the trajectory of excitation. The altered volume is then selectively 20 etched away. This results in a tube 126, as shown in Figure 6B. At higher densities of exposure, the remaining material becomes a random array of isolated fingers. The end of each tube 126 is then processed to form spatulae or spatulae are attached to the tubes.

Figure 7 illustrates another technique that may be utilized in accordance with 25 the invention. This embodiment relies upon the deposition of an etchable material on a substrate 130. Stalks 132 are then patterned and etched from the etchable material. The etched substrate may be coated with oxide and/or nitride layers. Alternately, polymer layers may be used as a coating. The polymer layers may be spin-cast, using materials, such as photoresist, polyimide, glass, or epoxy-based compounds. The 30 resultant stalks 132 are then seeded to form nanotubes 136, operating as spatulae.

Artificial spatulae may be formed using a glass micro-pipette drawn down to a narrow aperture (e.g., 500 nm) at an end. Liquid polymer is extruded through the hollow pipette and is then cured. Surface tension creates a hemispherical drop at the end of the pipette. Figure 8 illustrates this technique. In particular, the figure 5 illustrates a micro-pipette 150 with a liquid polymer 152 positioned therein to form a hemispherical drop 154.

Materials that can be applied to the micro-pipette include low viscosity ultra violet cure epoxy, uncured silicone rubber, or polyurethane resin. The hemisphere at the end of the micro-pipette can be flattened or embossed by pressing against a 10 polished surface. A flattened surface, such as the paddle structure of Figure 1E, with its larger contact area, has better adhesive properties than a sphere.

The single spatula pipette can be used as an embossing tool to make a nano-mold by plastically deforming a material, such as molten polystyrene. A large area mold (e.g., 20 by 20 microns) can be formed by either step-and-repeat embossing or 15 by making an array of pipettes and embossing a large pattern.

Figure 9 illustrates an array of pipettes used to form an embossing tool 160. The embossing tool 160 is applied to a polystyrene material 162 positioned on a substrate 164. This results in a patterned polystyrene surface 166.

Alternatively, a nano-channel glass, which consists of a large bundle of hollow 20 glass fibers, can be used. The nano-channel glass can be filled with a polymer, and then the glass can be dissolved in an acid.

Spatulae may also be formed by lithographically induced self construction. With this technique, electrostatic attraction is used to pull liquid through a mask, and thereby "sprout" spatulae. This process is shown in connection with Figures 10A- 25 10D.

Figure 10A illustrates a growing layer 170 positioned on a substrate 172. The growing layer may be molten polymer or thermoplastic. Spacers 174 are positioned on the substrate 172 and a mask 176 is constructed on the spacers 174. The growing layer 170 is electrostatically attracted to the upper mask layer 176, producing a set of 30 protrusions 178, as shown in Figure 10C. The resultant spatulae array is shown in Figure 10D.

Stalks and spatulae may also be formed from a mold using a nano-imprinting roller. This technique is shown in connection with Figures 11A-11B. While nano-imprinting techniques of the type shown in Figures 11A-11B have been used in the prior art, they have not been used to produce spatulae structures.

5       Figure 12 illustrates that a 2-layer photoresists can be formed with different resist exposure sensitivities, so that the upper layer forms, for example, 100 nm square plates that are supported by much longer and thinner pedestals. Standing-wave interference patterns can be used to expose and pattern features to fabricate large area arrays. Similar structures can be made with SiO<sub>x</sub> layers on silicon substrates by  
10 plasma etching.

Setae shafts may be fabricated using a sandwich of polymer layers. A polymer layer can include spin-cast polymer materials, such as photoresist, polyimide, glass, or epoxy-based compounds. A polymer layer can also include spray-deposited polymer materials, such as photoresist, polyimide, glass, or epoxy-based compounds.

15      Alternately, a polymer layer may be an ultra-violet curable epoxy.

Figure 13 illustrates a manipulator 200 formed in accordance with an embodiment of the invention. The manipulator 200 includes a beam 202 with a set of setae 26A-26D arranged in opposing pairs (e.g., 26A and 26C oppose one another, as do 26B and 26D). The beam 202 is pushed toward the substrate 204 to preload and  
20 spread the setae 26A-26D. The beam 202 is then pulled away from the substrate 204 to drag and pick-up the substrate 204. The beam 202 is pushed toward the substrate 204 to release the setae 26A-26D.

Those skilled in the art will recognize that the adhesive microstructures of the invention may be utilized in a variety of ways. For example, the technique of the  
25 invention can be used in pick and place micromanufacturing, micromanipulation, and microsurgery applications. For example, a seta can be attached to a micromanipulator to pick up a fiber optic, move it, and put it down again. Other uses include manipulating retinal prosthesis implants/explants, attaching to nerves during surgery, and pick and place of silicon wafers or disk drive components.

The setae of the invention may also be used as clutch mechanisms in micromachines. Since setae adhere in a directional manner, a seta could be used as a clutch mechanism similar to a ratchet, but on a smooth surface.

Other applications for the technique of the invention include: insect trapping, 5 tape, robot feet or treads, gloves/pads for climbing, gripping, etc., clean room processing tools, micro-optical manipulation that does not scar a surface and leaves no residue or scratches, micro-brooms, micro-vacuums, flake removal from wafers, optical location and removal of individual particles, climbing, throwing, and sticker toys, press-on fingernails, silent fasteners, a substrate to prevent adhesion on specific 10 locations, a broom to clean disk drives, post-it notes, band aids, semiconductor transport, clothes fasteners, and the like. In many of these applications, patches of spatula on a planar substrate are used, as opposed to patches of spatula positioned on a shaft.

The foregoing description, for purposes of explanation, used specific 15 nomenclature to provide a thorough understanding of the invention. However, it will be apparent to one skilled in the art that the specific details are not required in order to practice the invention. Thus, the foregoing descriptions of specific embodiments of the present invention are presented for purposes of illustration and description. They are not intended to be exhaustive or to limit the invention to the precise forms 20 disclosed, obviously many modifications and variations are possible in view of the above teachings. The embodiments were chosen and described in order to best explain the principles of the invention and its practical applications, to thereby enable others skilled in the art to best utilize the invention and various embodiments with various modifications as are suited to the particular use contemplated. It is intended 25 that the scope of the invention be defined by the following claims and their equivalents.

IN THE CLAIMS:

1 1. A method of forming an adhesive force, said method comprising the steps of:  
2 removing a seta from a living specimen;  
3 attaching said seta to a substrate; and  
4 applying said seta to a surface so as to establish an adhesive force between  
5 said substrate and said surface.

1 2. The method of claim 1 wherein said removing step includes the step of  
2 removing a seta from a gecko.

1 3. The method of claim 1 wherein said removing step includes the step of  
2 removing a seta from a living specimen selected from the group consisting of species  
3 of *Anolis*, skink, beetles, and kissing-bugs.

1 4. The method of claim 1 wherein said applying step includes the steps of:  
2 applying said seta to said surface with a force perpendicular to said surface;  
3 and  
4 pulling said seta with a force parallel to said surface so as to engage said  
5 adhesive force.

1 5. The method of claim 4 wherein said adhesive force is greater than the  
2 cumulative force of said applying and pulling steps.

1 6. A method of establishing a adhesive microstructure, said method comprising  
2 the steps of:  
3 applying a seta to a surface with a force perpendicular to said surface;  
4 pulling said seta with a force parallel to said surface so as to preload an  
5 adhesive force of said seta.

1 7. The method of claim 6 wherein said adhesive force is greater than the  
2 cumulative force of said applying and pulling steps.

1 8. A method of fabricating an adhesive microstructure, said method comprising  
2 the steps of:  
3 fabricating an array of shafts; and  
4 forming spatulae on said array of shafts.

1 9. The method of claim 8 wherein said fabricating step includes the step of  
2 constructing said array of shafts with a sandwich of nitride and oxide.

1 10. The method of claim 8 wherein said fabricating step includes the step of  
2 constructing said array of shafts with a sandwich of polymer layers.

1 11. The method of claim 10 wherein said constructing step includes the step of  
2 constructing said array of shafts with a sandwich of polymer layers, wherein a  
3 polymer layer is a spin-cast polymer material selected from the group consisting of  
4 photoresist, polyimide, glass, and epoxy-based compounds.

1 12. The method of claim 10 wherein said constructing step includes the step of  
2 constructing said array of shafts with a sandwich of polymer layers, wherein a  
3 polymer layer is a spray-deposited polymer material selected from the group  
4 consisting of photoresist, polyimide, glass, and epoxy-based compounds.

1 13. The method of claim 10 wherein said constructing step includes the step of  
2 constructing said array of shafts with a sandwich of polymer layers including ultra  
3 violet curable epoxies.

1 14. A fabricated microstructure, comprising:  
2 a shaft with a length of less than 500 microns, said shaft having a diameter of  
3 between 0.01 and 0.1 times said length of said shaft; and

4           an array of spatulae formed at an end of said shaft, said array of spatulae  
5   having a width of less than 10 microns, individual spatula of said spatulae including a  
6   curved segment of a sphere at a spatula terminal end.

1   15.   The fabricated microstructure of claim 14 wherein said shaft has a length of  
2   between approximately 10 and 100 microns.

1   16.   The fabricated microstructure of claim 14 wherein said shaft has a diameter of  
2   approximately 0.05 times said length of said shaft.

1   17.   The fabricated microstructure of claim 14 wherein said curved segment of a  
2   sphere at a spatula terminal end has a radius of approximately  $2\mu\text{m}$ .

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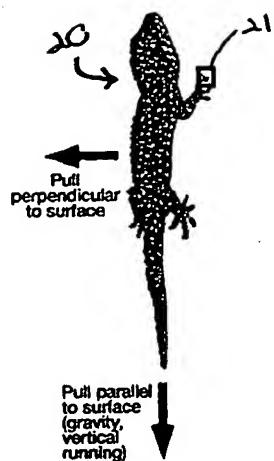


Fig. 1A

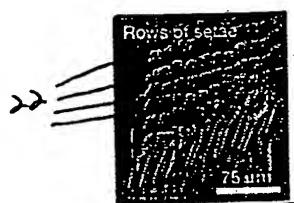


Fig. 1B

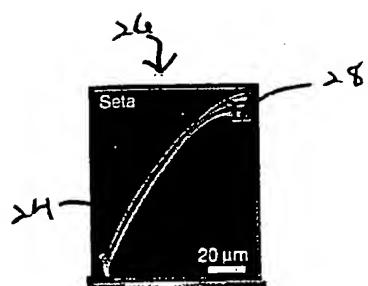


Fig. 1C

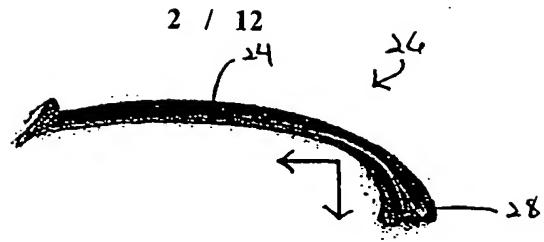


Fig. 1D

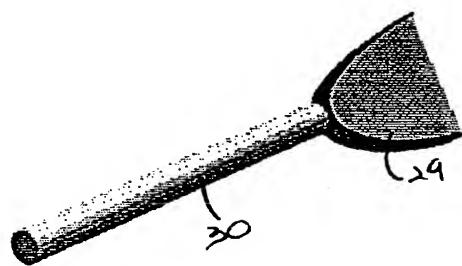


Fig. 1E

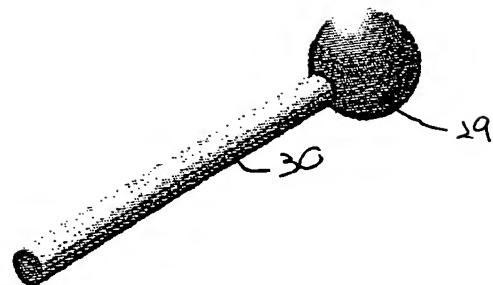


Fig. 1F

3 / 12

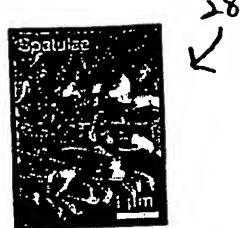


Fig. 1G

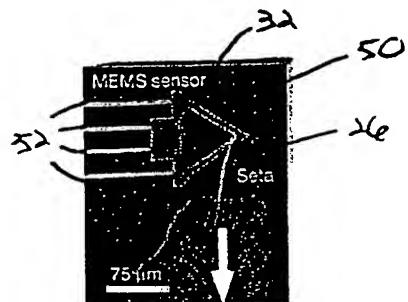


Fig. 1H

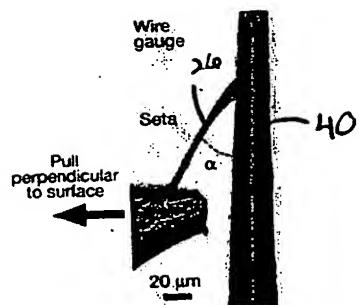


Fig. 1I

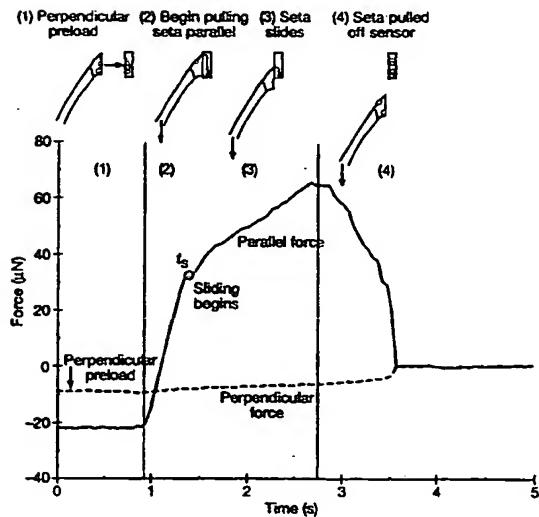


Fig. 2A

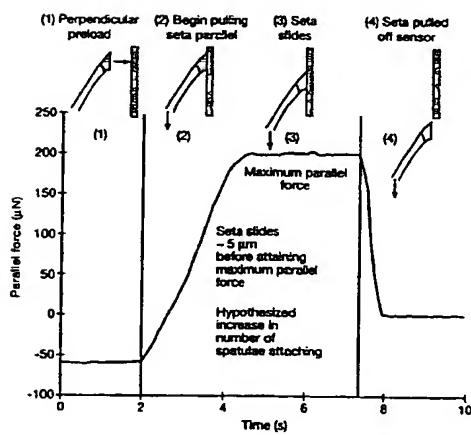


Fig. 2B

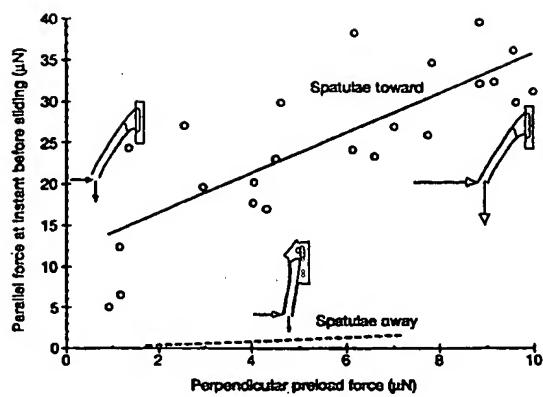


Fig. 3

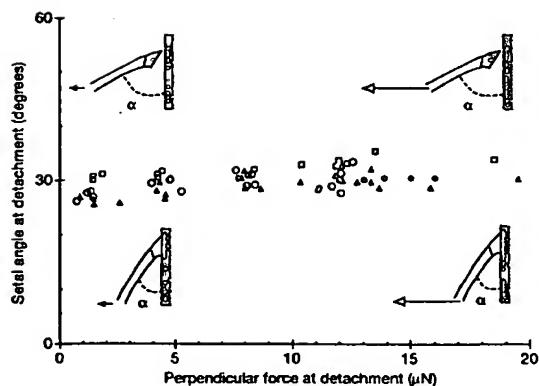


Fig. 4

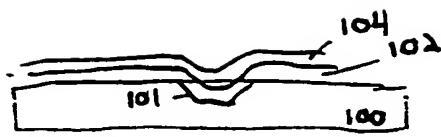


Fig. 5A



Fig. 5B

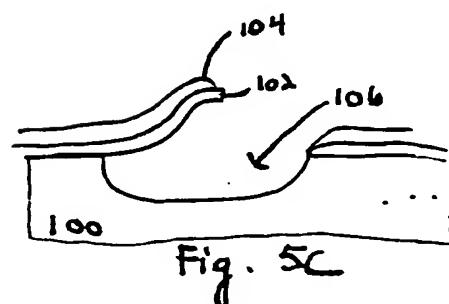


Fig. 5C

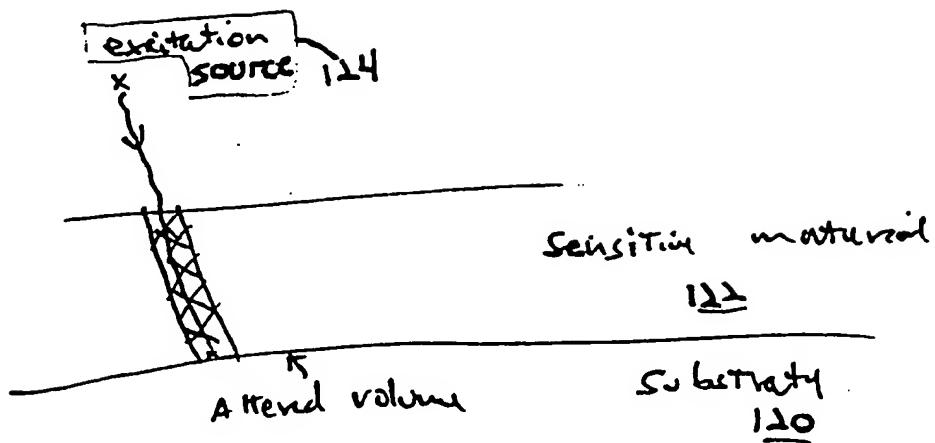


Fig. 6A

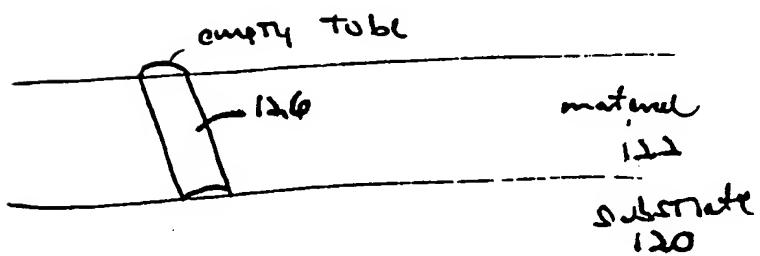


Fig. 6B

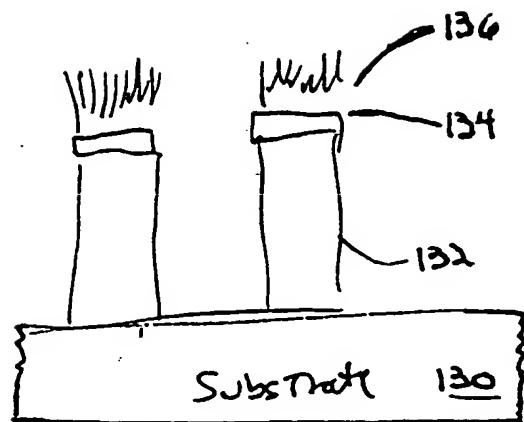


Fig. 7

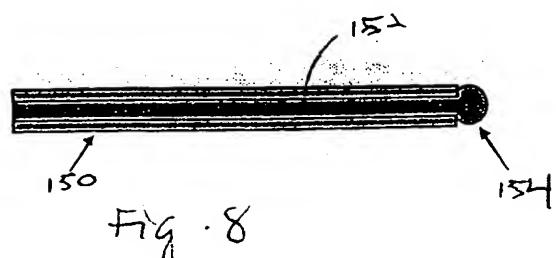


Fig. 8

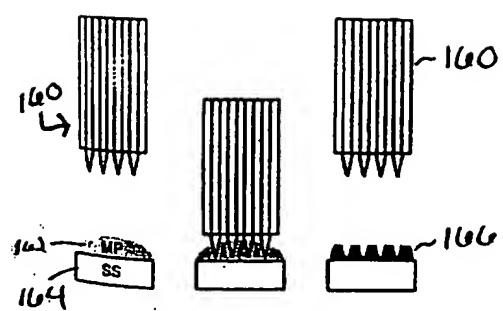


Fig. 9

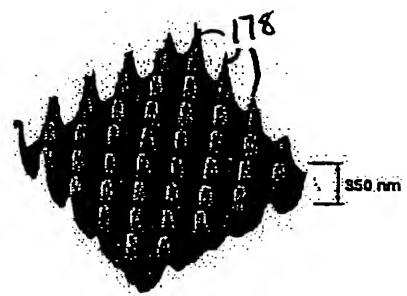
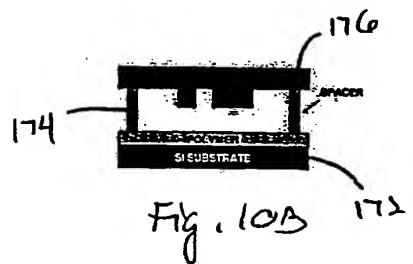
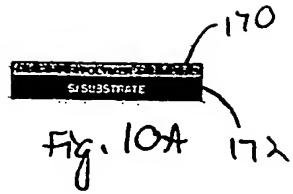


Fig. 10D

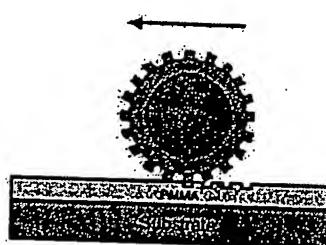


Fig. 11A

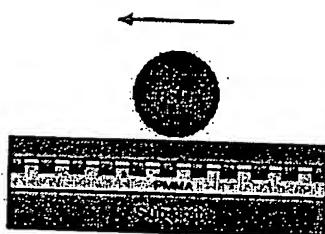


Fig. 11B

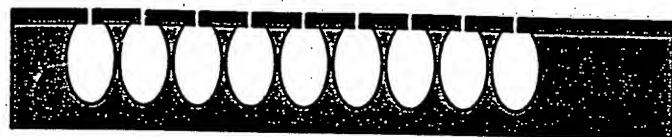


Fig. 12

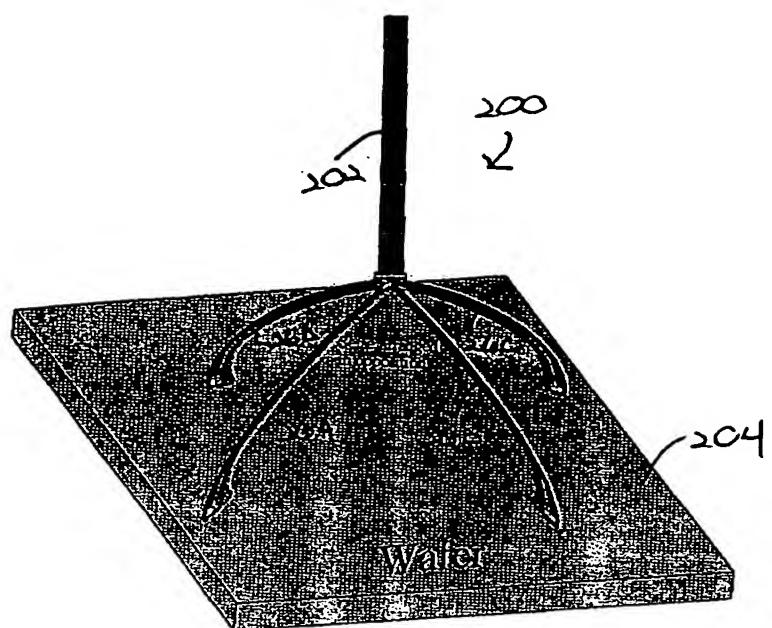


Fig. 13

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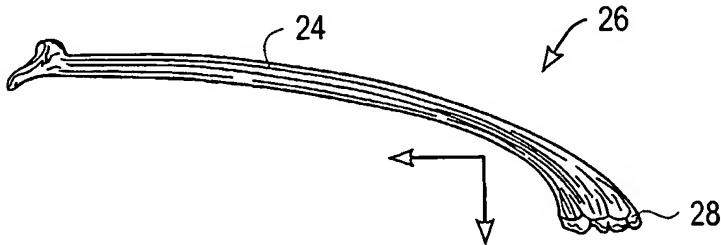
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(72) Inventors: FULL, Robert, J.; 2855 West Watson Court, Concord, CA 94518 (US). FEARING, Ronald, S.; 7010 Manila Avenue, El Cerrito, CA 94530 (US). KENNY, Thomas, W.; 132 Devonshire Boulevard, San Carlos, CA 94070 (US). AUTUMN, Kellar; 1919 SW Martha Street, Portland, OR 97201 (US).

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: ADHESIVE MICROSTRUCTURE AND METHOD OF FORMING SAME

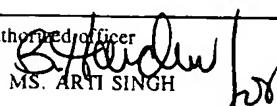
WO 01/49776 A3



(57) Abstract: A method of forming an adhesive force includes removing a seta from a living specimen, attaching the seta to a substrate, and applying the seta to a surface so as to establish an adhesive force between the substrate and the surface. The seta is applied to the surface with a force perpendicular to the surface. The seta is then pulled with a force parallel to the surface so as to preload the adhesive force of the seta.

# INTERNATIONAL SEARCH REPORT

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<b>A. CLASSIFICATION OF SUBJECT MATTER</b>																						
IPC(7) : Please See Extra Sheet. US CL : Please See Extra Sheet.																						
According to International Patent Classification (IPC) or to both national classification and IPC																						
<b>B. FIELDS SEARCHED</b>																						
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U.S. : Please See Extra Sheet.																						
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Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) <b>EAST</b>																						
<b>C. DOCUMENTS CONSIDERED TO BE RELEVANT</b>																						
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.																				
A	US 4,545,831A (ORNSTEIN) 08 October 1985. see entire document.	1-17N																				
A	US 5,843,657A (LIOTTA et al.) 01 December 1998, see entire document.	1-17																				
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Date of the actual completion of the international search  31 MAY 2001	Date of mailing of the international search report  <i>21 JUN 2001</i>																					
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B 32 B 31/14;  
C 12 N 5/00.

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427/4;  
428/16, 343;  
435/6.1, 325, 1.17;  
156/57, 61, 62.2, 68, 77, 241;  
424/81, 3.

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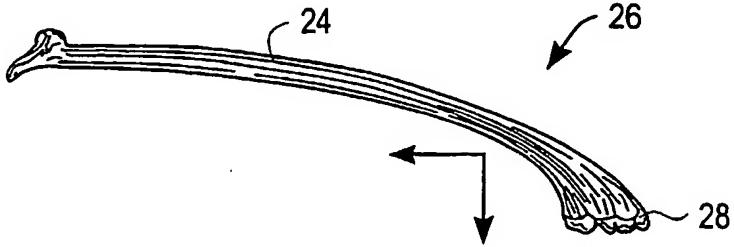
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(54) Title: ADHESIVE MICROSTRUCTURE AND METHOD OF FORMING SAME

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(57) Abstract: A method of forming an adhesive force includes removing a seta from a living specimen, attaching the seta to a substrate, and applying the seta to a surface so as to establish an adhesive force between the substrate and the surface. The seta is applied to the surface with a force perpendicular to the surface. The seta is then pulled with a force parallel to the surface so as to preload the adhesive force of the seta.

## **ADHESIVE MICROSTRUCTURE AND METHOD OF FORMING SAME**

This application claims priority to the provisional patent application entitled, "Non-Interlocking Dry Adhesive Microstructure and Method of Forming Same," Serial Number 60/172,731, filed December 20, 1999.

This invention was made with Government support under Grant (Contract) No. N00014-98-C0183 awarded by DARPA through a subcontract from I.S. Robotics. The Government has certain rights to this invention.

### **BRIEF DESCRIPTION OF THE INVENTION**

This invention relates generally to the fabrication and utilization of micron-scale structures. More particularly, this invention relates to a non-interlocking adhesive microstructure.

### **BACKGROUND OF THE INVENTION**

There is an ongoing need for improved adhesives. Improved adhesives have applications ranging from everyday aspects of life (e.g., tape, fasteners, and toys) to high technology (e.g., removal of microscopic particles from semiconductor wafers, transporting fiber optic devices, and assembly of sub-mm mechanisms, particularly those including micro-fabricated components, or components that cannot tolerate grippers, adhesives, or vacuum manipulators).

Adhesive mechanisms in nature have been studied, but have not been fully understood or exploited. For example, Geckos are exceptional in their ability to rapidly climb up smooth vertical surfaces. The mechanism of adhesion used in Geckos, *Anolis* lizards, some skinks, and some insects, has been debated for nearly a 5 century.

While some prior work has identified the morphology of seta used by Geckos and other insects, this prior work does not identify how the seta operates. In addition, this prior work fails to identify how to use a seta to perform useful work.

It would be highly desirable to identify and exploit the adhesive force 10 mechanism utilized by Geckos and other insects. Such information could result in the utilization of new adhesive microstructures and the fabrication of such structures.

### SUMMARY OF THE INVENTION

The invention includes a method of forming an adhesive force. The method 15 includes the steps of removing a seta from a living specimen, attaching the seta to a substrate, and applying the seta to a surface so as to establish an adhesive force between the substrate and the surface.

The invention also includes a method of establishing a adhesive 20 microstructure. The method includes the step of applying a seta to a surface with a force perpendicular to the surface. The seta is then pulled with a force parallel to the surface so as to preload an adhesive force of the seta.

The invention also includes a method of fabricating an adhesive 25 microstructure. The fabrication technique includes fabricating an array of shafts and then forming spatulae on the array of shafts.

The invention also includes a fabricated microstructure with a shaft having a length of less than 500 microns. The shaft has a diameter of between 0.01 and 0.1 times the length of the shaft. An array of spatulae is formed at an end of the shaft. The array of spatulae has a width of less than 10 microns. Individual spatula of the spatulae include a terminal end with an extended surface, such as a paddle, a curved 30 segment of a sphere, or a flattened segment of a sphere.

**BRIEF DESCRIPTION OF THE DRAWINGS**

For a better understanding of the invention, reference should be made to the following detailed description taken in conjunction with the accompanying drawings, in which:

5 FIGURE 1A illustrates preloading operations performed in accordance with an embodiment of the invention.

FIGURE 1B illustrates rows of setae used in accordance with the invention.

FIGURE 1C illustrates a single seta used in accordance with the invention.

10 FIGURE 1D is an enlarged view of a single seta used in accordance with an embodiment of the invention.

FIGURE 1E is an enlarged view of a single extended surface spatula on a spatula stalk, in accordance with an embodiment of the invention.

FIGURE 1F is an enlarged view of a single extended surface spatula on a spatula stalk, in accordance with an embodiment of the invention.

15 FIGURE 1G illustrates an array of spatulae formed at the end of a shaft to form seta used in accordance with the invention.

FIGURE 1H illustrates a system to measure adhesive force achieved in accordance with the invention.

20 FIGURE 1I is another illustration of a system to measure adhesive force achieved in accordance with the invention.

FIGURES 2A-2B illustrate different forces, as a function of time, associated with the loading and adhesion operation of a structure of the invention.

FIGURE 3 illustrates perpendicular preload force associated with an embodiment of the invention.

25 FIGURE 4 illustrates perpendicular force during detachment of a structure utilized in accordance with the invention.

FIGURES 5A-5C illustrate the fabrication of an array of setae with spatula structures in accordance with an oxide/nitride fabrication process utilized in accordance with an embodiment of the invention.

FIGURES 6A-6B illustrate the fabrication of an array of setae in accordance with an excitation source process utilized in accordance with an embodiment of the invention.

5 FIGURE 7 illustrates the fabrication of an array of setae with spatula structures in accordance with a stalk and seeding process utilized in accordance with an embodiment of the invention.

FIGURE 8 illustrates the fabrication of a single spatula using a micro-pipette.

FIGURE 9 illustrates an embossing tool used to form a spatulae mold for use in accordance with an embodiment of the invention.

10 FIGURES 10A-10D illustrate lithographically induced self-construction of spatulae in accordance with an embodiment of the invention.

FIGURES 11A-11B illustrate a roller nano-imprinting technique that may be used to form spatulae in accordance with an embodiment of the invention.

15 FIGURE 12 illustrates a two-layer photoresist fabrication technique that may be used in accordance with an embodiment of the invention.

FIGURE 13 illustrates a setae-based manipulator that may be used in accordance with an embodiment of the invention.

Like reference numerals refer to corresponding parts throughout the drawings.

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## DETAILED DESCRIPTION OF THE INVENTION

The invention is directed toward the use of micron scale structures to achieve adhesion. In particular, the invention uses a seta structure. The seta structure has a shaft. Positioned at the end of the shaft is a spatula or an array of spatulae. Adhesion is produced as the spatula or array of spatulae produce intimate contact with a surface.

25 In general, the shaft is between 1 and 500 microns long, preferably approximately 10 to 100 microns long. The diameter of the shaft is preferably between 0.01 and 0.1 times the length of the shaft, preferably approximately 0.05 times the length of the shaft.

30 The terminal end of the shaft has at least one spatula. Preferably, the terminal end of the shaft has between 1 and 1000 spatulae. The array of spatulae is preferably less than 10 microns wide, preferably approximately 1 micron wide. Preferably, each

spatula of the array of spatulae has an extended surface at its terminal end. The extended surface may be in the form of a paddle or a curved segment of a sphere, as shown below.

The structure of the invention is modeled from structures found in nature, such 5 as the seta found on the foot of a Tokay gecko (*Gekko gecko*). Many species of gecko (e.g., clade Gekkonoidea), species of *Anolis*, and several skink species have adhesive setae that may also be used in accordance with the invention. In addition, beetles and kissing-bugs have setae that may be used in accordance with the invention. The invention is implemented with natural or fabricated setae, as discussed below.

10 Examples of seta structures found in nature follow. The seta of a Tokay Gecko has a stalk (shaft) diameter of  $5\mu$ , a stalk height of  $110\mu$ , a tip (spatulae) length of  $0.2\mu$ , a tip width of  $0.2\mu$ , and between 100-1000 tips, where the total tip area per stalk is 2 to  $20\mu^2$ . *Anolis cuvieri* has a stalk diameter of  $0.5\mu$ , a stalk height of  $22\mu$ , a tip length of  $0.6\mu$ , a tip width of  $0.7\mu$ , and between 100-1000 tips, where the 15 total tip area per stalk is 2 to  $20\mu^2$ . *Prasinohaema virens* (skink) has a stalk diameter of  $2\mu$ , a stalk height of  $26\mu$ , a tip length of  $6\mu$ , a tip width of  $7\mu$ , and between 100-1000 tips, where the total tip area per stalk is approximately  $20\mu^2$ .

By way of example, Figure 1A illustrates a Tokay gecko 20 with terminal 20 limbs 21 that have naturally occurring setae. The live gecko 20 is restrained. The cuticular layer of a portion of a terminal limb (e.g., a toe) 21 is removed. This operation, analogous to cutting hair, allows the gecko to harmlessly regenerate its setae. It has been demonstrated that hundreds or thousands of setae can be easily harvested without sacrificing the living being from which the setae are removed.

After removal, the cuticular surface is scraped to break off individual seta, 25 preferably at the base of the shaft of the seta. Figure 1B illustrates rows 22 of setae associated with the gecko 20. Figure 1C illustrates the shaft 24 of a single seta 26. The figure also illustrates the spatulae 28 positioned at the end of the shaft 24.

Figure 1D is an enlarged view of a single seta 26. The figure illustrates that the shaft 24 is roughly perpendicular to the spatulae 28.

30 Figure 1E is an enlarged view of a single spatula 29 on a spatula stalk 30. The spatula stalk 30 may be the shaft 24 or a separate tendril extending from the shaft 24.

Preferably, each spatula 29 has an extended surface. In Figure 1E, the extended surface is in the form of a paddle structure. In Figure 1F, the extended surface is in the form of a sphere. Figure 1G is an enlarged view of an array of spatulae 28.

The realization of large adhesive forces by the setae is contingent upon preload operations performed in accordance with the invention. Detachment of the setae occurs at a characteristic angle, as specified in accordance with the invention. Figure 1H illustrates a substrate (i.e., a sensor 32) that was used to characterize these forces. Figure 1I illustrates the characteristic angle ( $\alpha$ ) for detaching setae of the invention. The characteristic angle ( $\alpha$ ) is formed between the seta 26 and a surface 40 to which the seta is attached.

The inventors have identified that the adhesive force of a seta depends upon its three-dimensional orientation (spatulae pointing toward or away from the surface) and the extent to which the seta is preloaded (pushed into and pulled along the surface) during initial contact. Contacting the surface with the seta in a direction other than with spatulae projecting toward the surface resulted in forces less than 0.3  $\mu$ N when the seta was pulled away perpendicular to the surface. A pull parallel to the surface showed that the force produced by the inactive, non-spatular region increased with normal or perpendicular force, typical of a material with a coefficient of friction equal to 0.2. By contrast, when the active spatular region was projecting toward the surface, force increased by 20 to 60-fold. The force resulting from pulling the seta parallel to the surface during attachment increased when setae were first pushed toward the surface, providing a perpendicular preloading force. This initial perpendicular force need not be maintained during the subsequent pull. Setal force parallel to the surface increased linearly with the perpendicular preloading force.

Experiments in which seta were pulled away from the surface of a wire demonstrated that perpendicular preloading alone is insufficient to prevent the seta from being dislodged easily. Seta that were first pushed into the surface and then pulled parallel to it developed over ten times the force (13.6  $\mu$ N  $\pm$  2.6 SD; N = 17) upon being pulled away from the surface than those having only a perpendicular preload (0.6  $\mu$ N  $\pm$  0.7 SD; N = 17). The largest parallel forces were observed only following a few microns of sliding. The results of preloading on setal force

production suggest that a small perpendicular preloading force in concert with a rearward displacement or parallel preload may be necessary to "engage" adhesion. Preloading is believed to increase the number of spatulae contacting the surface.

The orientation of the setae is also important in detachment. The force produced when a seta was pulled away from the surface was not significantly differently from the force measured during a pull parallel to the surface if the same perpendicular preload was given. However, it has been identified that setae detached at a similar angle ( $30.6^\circ \pm 1.8$  SD; N = 17) and force when pulled away from the sensor's surface. To check for the presence of a critical angle of detachment, the perpendicular force was held constant, while the setal angle was progressively increased until detachment. Setal angle at detachment changed by only 15% over a range of perpendicular forces. Thus, the invention utilizes a detachment angle of between  $35^\circ$  and  $25^\circ$ , preferably approximately  $30^\circ$ . The detachment angle values are based upon the disclosed seta structure in which the shaft of the seta is roughly perpendicular to the spatular surface, as shown in Figure 1D. Change in the orientation of the setae and perhaps even the geometry of the spatulae may facilitate detachment.

The foot of a Tokay gecko (*Gekko gecko*) holds approximately 5000 setae  $\text{mm}^{-2}$  and can produce 10 N of adhesive force with approximately  $100 \text{ mm}^2$  of pad area. Therefore, each seta should produce an average force of  $20 \mu\text{N}$  and an average stress of  $0.1 \text{ N mm}^{-2}$  (~1 atm). The actual magnitudes are probably greater, since it is unlikely that all setae adhere simultaneously.

The foregoing information is more fully appreciated in connection with specific operations performed in accordance with the invention. An isolated seta, secured by the technique discussed above, was glued to a substrate (e.g., to the end of a #2 insect pin) with epoxy (e.g., 5-MINUTE EPOXY sold by TTWDevcon, Danvers, MA). The pin had a diameter of approximately 15  $\mu\text{m}$ . To prevent the epoxy from creeping up the stalk of the seta, which might change the mechanical property of the specimen, the epoxy is preferably precured for approximately 1 minute before applying it to the specimen. All setae were oriented such that the active surface was

approximately perpendicular to the axis of the pin. All preparations were completed under a compound microscope.

Force production by single, isolated seta during attachment was measured using a micromachined, dual-axis, piezoresistive sensor 32 of the type illustrated in 5 Figure 1H. The following discussion provides information with respect to the sensor 32. U.S. Patent 5,959,200 describes a sensor of the type described herein. The sensor 32 does not form a part of the invention, rather it is merely used to obtain the performance results described below.

The cantilever sensor 32 of Figure 1H was fabricated on a single-crystalline 10 silicon wafer. The cantilever 32 has two independent force sensors, each with one predominant direction of compliance. The perpendicular force sensor consists of a thin triangular probe 50. The parallel force sensor is composed of four long slender ribs 52. A special 45° oblique ion implantation allowed piezoresistive and conductive regions to be implanted on both the parallel and perpendicular surfaces 15 simultaneously. Forces applied to the tip of the sensor were resolved into these two orthogonal directions (parallel and perpendicular), and were measured by the changes in resistance of the piezoresistors. Since this device was originally designed for Atomic Force Microscope data storage applications, each of these cantilever devices had a sharp tip near the vertex of its triangular probe. For the gecko setae adhesion 20 measurement, the back-side of this device was used to provide a smooth surface for setal adhesion.

Each seta 26 was brought in contact with the sensor 32 by applying a small preload perpendicular to the surface to increase contact and induce adhesion. To 25 determine the effect of preload force on submaximal parallel force, preload force was varied when setae were attached to the tip of the sensor, as shown in Figure 3, which is discussed below. To measure maximal parallel force, the base of the triangular probe was used. Using the base increased area of contact, but did not allow for simultaneous measurement of preload forces. Sensor signals were taken while the seta was being pulled parallel to the surface by a piezoelectric manipulator at a rate of 30 ~5  $\mu\text{m sec}^{-1}$ . Sensor signals were amplified and filtered through a 300-Hz low-pass filter, and then digitized at 100 Hz using a 16-bit data acquisition card (LabView™ on

a PC). The collected data (in volts) were converted to deflections of the sensor through calibration constants, and multiplied by the stiffness of the cantilever to obtain force values.

Breaking or detachment force was defined as the maximal force a seta could exert perpendicular, or normal, to a surface immediately before it released. This value was determined for individual seta by measuring the amount it could displace a force gauge made from a 4.7 mm aluminum bonding wire with 25  $\mu\text{m}$  nominal diameter (American Fine Wire Corp., Selma, AL; the wire 40 is shown in Figure 1I). To maximize contact area of the active surface of the seta to the wire, a 50  $\mu\text{m} \times 100 \mu\text{m}$  section of the wire tip was flattened. The proximal end of the wire was fixed with epoxy onto a brass stub. The active surface of the seta was pressed against the flattened wire, producing a known perpendicular preload ( $1.6 \pm 0.25 \mu\text{N}$ ; mean $\pm$ SD). The force was measured using two different methods of detachment: (1) the seta was pulled normal to the wire; and (2) the insect pin was displaced  $19.7 \pm 3.45 \mu\text{m}$  along the wire to produce an additional parallel preload on the seta before pulling perpendicular or normal to the wire.

In all trials, detachment force was calculated from the maximum displacement of the wire pulled by the seta. All sequences were recorded with a video camera (e.g., a CCD camera sold by SONY) and digitized to a computer (e.g., an APPLE, MACINTOSH) using a video editing system (e.g., from MEDIA 100 Inc., Marlboro, MA). The initial position of the wire, the angle of the seta with respect to the wire and the position of the wire at the point of separation were recorded and analyzed using image analysis software (e.g., NIH-Image software). The amount of deflection in the force gauge was converted to adhesion force after the force gauge was calibrated against standard weights.

The results of these operations are shown in Figures 2-4. Figure 2A illustrates forces associated with a perpendicular preload and a subsequent parallel pulling performed in accordance with the invention. As shown in Figure 2A, setal adhesive force parallel to the surface increased linearly until the seta began to slide off the edge of the sensor at time  $t_s$ . If the seta was allowed to slide approximately 5  $\mu\text{m}$  along the sensor's surface, a distance imperceptible at the level of the foot, adhesive force

continued to increase, as shown in Figure 2B. The maximum adhesive force of single seta averaged  $194 \mu\text{N} \pm 25 \text{ SD}$  ( $N = 28$ ), nearly 10-fold greater than predicted from whole animal estimates.

The single-seta force measurements suggest that if all setae were

5 simultaneously and maximally attached, a single foot of a gecko could produce 100 N of adhesive force (-10 arm). Stated another way, the foot of a gecko could generate maximum observed force (10 N) with only 10% of its setae maximally attached.

The maximum force developed by a given seta could not be predicted from molecular interactions or microscopic anatomy alone. Setal force depended on its

10 three-dimensional orientation (spatulae pointing toward or away from the surface) and the extent to which the hair was preloaded (i.e., pushed into and pulled along the surface) during initial contact. Contacting the surface with the seta in a direction other than with spatulae projecting toward the surface resulted in forces less than  $0.3 \mu\text{N}$  when the seta was pulled away perpendicular to the surface. A pull parallel to the

15 surface showed that the force produced by the inactive, non-spatular region increased with normal or perpendicular force, typical of a material with a coefficient of friction equal to 0.25, see Figure 3. By contrast, when the active spatular region was projecting toward the surface, force increased by 20 to 60-fold. The force resulting from pulling the seta parallel to the surface during attachment increased when setae

20 were first pushed toward the surface providing a perpendicular preloading force, shown in Figure 2A. This initial perpendicular force need not be maintained during the subsequent pull. Setal force parallel to the surface increased linearly with the perpendicular preloading force, as shown in Figure 3. Experiments in which seta were pulled away from a surface (e.g., surface 40, a wire in Figure 1F) demonstrated

25 that perpendicular preloading alone is insufficient to prevent the seta from being dislodged easily. Seta that were first pushed into the surface and then pulled parallel to it developed over ten times the force ( $13.6 \mu\text{N} \pm 2.6 \text{ SD}$ ;  $N = 17$ ) upon being pulled away from the surface than those having only a perpendicular preload ( $0.6 \mu\text{N} \pm 0.7 \text{ SD}$ ;  $N = 17$ ). The largest parallel forces were observed only following a few microns

30 of sliding, as shown in Figure 2B.

The results of preloading on setal force production support the hypothesis that a small perpendicular preloading force in concert with a rearward displacement or parallel preload may be necessary to "engage" adhesion. Since the tips of the setae are directed rearwards away from the toenail, preloading may increase the number of 5 spatulae contacting the surface.

The orientation of the setae also appears to be important in detachment during locomotion. The force produced when a seta was pulled away from the surface was not significantly different from the force measured during a pull parallel to the surface if the same perpendicular preload was given. However, it was identified that setae 10 detached at a similar angle ( $30.6^\circ \pm 1.8$  SD; N = 17) when pulled away from the wire sensor's surface. To check for the presence of a critical angle of detachment, perpendicular force was held constant, while the setal angle ( $\alpha$ ; Figure 1F) progressively increased until detachment. Setal angle at detachment changed by only 15% over a range of perpendicular forces, as shown in Figure 4. This observation is 15 consistent with an adhesive model where sliding stops when pulling at greater than the critical setal angle and hence stress can increase at a boundary, causing fracture of the contact. Change in the orientation of the setae and perhaps even the geometry of the spatulae may facilitate detachment.

It has long been known that geckos peel the tips of their toes away from a 20 smooth surface during running. Toe peeling may have two effects. First, it may put an individual seta in an orientation or at a critical angle that aids in its release. Second, toe peeling concentrates the detachment force on only a small subset of all attached setae at any instant. The toe peeling behavior is analogous to the technique used by humans to remove a piece of tape from a surface.

25 The direct setal force measurements are consistent with the hypothesis that adhesion in geckos is the result of intermolecular forces. The simple models available can only give the most approximate estimates of setal force production. If it is assumed that the tip of a spatula is a curved segment of a sphere (radius,  $R = 2\mu\text{m}$ ) and is separated by a small distance from a large, flat surface where van der Waals 30 forces become significant (atomic gap distance,  $D \approx 0.3$  nm), then setal force =  $AR / 6D^2$ , where A is the material dependent Hamaker constant taken to be  $10^{-19} \text{ J}^{10}$ . This

estimate puts the van der Waals force for a spatula to be about  $0.4 \mu\text{N}$ . Since the number of spatula per seta varies from 100 to 1000, setal force estimates range from 40 to  $400 \mu\text{N}$ .

Earlier experimental support for the van der Waals hypothesis comes from the 5 observation that adhesive force of a whole gecko increases with increasing surface energy of the substrate. In addition, the rejection of alternative mechanisms such as suction, electrostatics, friction, microinterlocking, and wet adhesion, has been attempted. Adhesion experiments carried out in a vacuum and the disclosed measurements of greater than one atmosphere of adhesion pressure strongly suggest 10 that suction is not involved. Experiments using X-ray bombardment eliminates electrostatic attraction as a mechanism necessary for setal adhesion, since the setae can still adhere in ionized air. Microinterlocking could function as a secondary mechanism, but the ability of geckos to adhere to polished glass shows that irregularities on the scale of the spatulae are not necessary for adhesion. The findings 15 herein do not support a friction mechanism because the cantilever's surface is smooth (surface roughness less than or equal to  $2.5 \text{ nm}$ ) and the coefficient of friction of the setal keratin on silicon is low ( $\mu = 0.25$ ; Fig. 3; dashed line). Capillary adhesion or glue are not likely mechanisms, since skin glands are not present on the feet of lizards. The mechanism of adhesion may involve a thin layer of water, or adsorbed water 20 molecules on the seta and/or substrate.

Van der Waals forces are extremely weak at greater than atomic distance gaps, and require intimate contact between the adhesive and the surface. Polymeric adhesives such as tape are soft, and are able to deform sufficiently for intimate contact over a relatively large surface area. The feet of a Tokay gecko (*Gekko gecko*) contain 25 approximately one billion spatulae that appear to provide a sufficiently large surface area in close contact with the substrate for adhesion to be the result of van der Waals forces.

As previously indicated, the invention may be used in connection with setae harvested from a live specimen. Alternately, the techniques of the invention may be 30 used in connection with fabricated setae. Those skilled in the art will recognize a number of techniques that may be used to fabricate setae in accordance with the

invention. For example, the devices may be fabricated through an oxide/nitride process, as shown in Figures 5A-5C.

Initially, a recess is etched in a semiconductor substrate. Figure 5A illustrates a recess 101 formed in a semiconductor substrate 100. Nitride and oxide layers are 5 then deposited on the substrate 100. Figure 5A illustrates a nitride layer 102 and an oxide layer 104. The surface is then patterned and etched, resulting in the structure of Figure 5B.

Afterwards, the underlying substrate 100 is etched, resulting in a well 106, as shown in Figure 5C. At this point, the stress difference between the oxide and nitride 10 layers causes the structure to curl from the plane defined by the substrate 100, thereby forming a shaft structure. The end of the shaft may then be roughened to form spatulae. For example, the spatulae may be formed by wet etching, radiation, plasma roughening, electro-chemical etching, and the like. Alternately, a separate spatulae may be affixed to the shaft. Techniques for fabricating spatulae are discussed below.

15 Another technique that may be utilized in accordance with the invention exploits an excitation source. As shown in Figure 6A, a sensitive material 122 is formed on a substrate 120. An excitation source 124 is used to apply excitation energy to the sensitive material 122. The deep-penetrating excitation alters the volume along the trajectory of excitation. The altered volume is then selectively 20 etched away. This results in a tube 126, as shown in Figure 6B. At higher densities of exposure, the remaining material becomes a random array of isolated fingers. The end of each tube 126 is then processed to form spatulae or spatulae are attached to the tubes.

Figure 7 illustrates another technique that may be utilized in accordance with 25 the invention. This embodiment relies upon the deposition of an etchable material on a substrate 130. Stalks 132 are then patterned and etched from the etchable material. The etched substrate may be coated with oxide and/or nitride layers. Alternately, polymer layers may be used as a coating. The polymer layers may be spin-cast, using materials, such as photoresist, polyimide, glass, or epoxy-based compounds. The 30 resultant stalks 132 are then seeded to form nanotubes 136, operating as spatulae.

Artificial spatulae may be formed using a glass micro-pipette drawn down to a narrow aperture (e.g., 500 nm) at an end. Liquid polymer is extruded through the hollow pipette and is then cured. Surface tension creates a hemispherical drop at the end of the pipette. Figure 8 illustrates this technique. In particular, the figure 5 illustrates a micro-pipette 150 with a liquid polymer 152 positioned therein to form a hemispherical drop 154.

Materials that can be applied to the micro-pipette include low viscosity ultra violet cure epoxy, uncured silicone rubber, or polyurethane resin. The hemisphere at the end of the micro-pipette can be flattened or embossed by pressing against a 10 polished surface. A flattened surface, such as the paddle structure of Figure 1E, with its larger contact area, has better adhesive properties than a sphere.

The single spatula pipette can be used as an embossing tool to make a nano-mold by plastically deforming a material, such as molten polystyrene. A large area mold (e.g., 20 by 20 microns) can be formed by either step-and-repeat embossing or 15 by making an array of pipettes and embossing a large pattern.

Figure 9 illustrates an array of pipettes used to form an embossing tool 160. The embossing tool 160 is applied to a polystyrene material 162 positioned on a substrate 164. This results in a patterned polystyrene surface 166.

Alternatively, a nano-channel glass, which consists of a large bundle of hollow 20 glass fibers, can be used. The nano-channel glass can be filled with a polymer, and then the glass can be dissolved in an acid.

Spatulae may also be formed by lithographically induced self construction. With this technique, electrostatic attraction is used to pull liquid through a mask, and thereby "sprout" spatulae. This process is shown in connection with Figures 10A- 25 10D.

Figure 10A illustrates a growing layer 170 positioned on a substrate 172. The growing layer may be molten polymer or thermoplastic. Spacers 174 are positioned on the substrate 172 and a mask 176 is constructed on the spacers 174. The growing layer 170 is electrostatically attracted to the upper mask layer 176, producing a set of 30 protrusions 178, as shown in Figure 10C. The resultant spatulae array is shown in Figure 10D.

Stalks and spatulae may also be formed from a mold using a nano-imprinting roller. This technique is shown in connection with Figures 11A-11B. While nano-imprinting techniques of the type shown in Figures 11A-11B have been used in the prior art, they have not been used to produce spatulae structures.

5       Figure 12 illustrates that a 2-layer photoresists can be formed with different resist exposure sensitivities, so that the upper layer forms, for example, 100 nm square plates that are supported by much longer and thinner pedestals. Standing-wave interference patterns can be used to expose and pattern features to fabricate large area arrays. Similar structures can be made with SiO<sub>x</sub> layers on silicon substrates by  
10 plasma etching.

Setae shafts may be fabricated using a sandwich of polymer layers. A polymer layer can include spin-cast polymer materials, such as photoresist, polyimide, glass, or epoxy-based compounds. A polymer layer can also include spray-deposited polymer materials, such as photoresist, polyimide, glass, or epoxy-based compounds.

15     Alternately, a polymer layer may be an ultra-violet curable epoxy.

Figure 13 illustrates a manipulator 200 formed in accordance with an embodiment of the invention. The manipulator 200 includes a beam 202 with a set of setae 26A-26D arranged in opposing pairs (e.g., 26A and 26C oppose one another, as do 26B and 26D). The beam 202 is pushed toward the substrate 204 to preload and  
20 spread the setae 26A-26D. The beam 202 is then pulled away from the substrate 204 to drag and pick-up the substrate 204. The beam 202 is pushed toward the substrate 204 to release the setae 26A-26D.

Those skilled in the art will recognize that the adhesive microstructures of the invention may be utilized in a variety of ways. For example, the technique of the  
25 invention can be used in pick and place micromanufacturing, micromanipulation, and microsurgery applications. For example, a seta can be attached to a micromanipulator to pick up a fiber optic, move it, and put it down again. Other uses include manipulating retinal prosthesis implants/explants, attaching to nerves during surgery, and pick and place of silicon wafers or disk drive components.

The setae of the invention may also be used as clutch mechanisms in micromachines. Since setae adhere in a directional manner, a seta could be used as a clutch mechanism similar to a ratchet, but on a smooth surface.

Other applications for the technique of the invention include: insect trapping,

5 tape, robot feet or treads, gloves/pads for climbing, gripping, etc., clean room processing tools, micro-optical manipulation that does not scar a surface and leaves no residue or scratches, micro-brooms, micro-vacuums, flake removal from wafers, optical location and removal of individual particles, climbing, throwing, and sticker toys, press-on fingernails, silent fasteners, a substrate to prevent adhesion on specific

10 locations, a broom to clean disk drives, post-it notes, band aids, semiconductor transport, clothes fasteners, and the like. In many of these applications, patches of spatula on a planar substrate are used, as opposed to patches of spatula positioned on a shaft.

The foregoing description, for purposes of explanation, used specific

15 nomenclature to provide a thorough understanding of the invention. However, it will be apparent to one skilled in the art that the specific details are not required in order to practice the invention. Thus, the foregoing descriptions of specific embodiments of the present invention are presented for purposes of illustration and description. They are not intended to be exhaustive or to limit the invention to the precise forms

20 disclosed, obviously many modifications and variations are possible in view of the above teachings. The embodiments were chosen and described in order to best explain the principles of the invention and its practical applications, to thereby enable others skilled in the art to best utilize the invention and various embodiments with various modifications as are suited to the particular use contemplated. It is intended

25 that the scope of the invention be defined by the following claims and their equivalents.

IN THE CLAIMS:

1    1.    A method of forming an adhesive force, said method comprising the steps of:  
2         removing a seta from a living specimen;  
3         attaching said seta to a substrate; and  
4         applying said seta to a surface so as to establish an adhesive force between  
5         said substrate and said surface.

1    2.    The method of claim 1 wherein said removing step includes the step of  
2         removing a seta from a gecko.

1    3.    The method of claim 1 wherein said removing step includes the step of  
2         removing a seta from a living specimen selected from the group consisting of species  
3         of *Anolis*, skink, beetles, and kissing-bugs.

1    4.    The method of claim 1 wherein said applying step includes the steps of:  
2         applying said seta to said surface with a force perpendicular to said surface;  
3         and  
4         pulling said seta with a force parallel to said surface so as to engage said  
5         adhesive force.

1    5.    The method of claim 4 wherein said adhesive force is greater than the  
2         cumulative force of said applying and pulling steps.

1    6.    A method of establishing a adhesive microstructure, said method comprising  
2         the steps of:  
3         applying a seta to a surface with a force perpendicular to said surface;  
4         pulling said seta with a force parallel to said surface so as to preload an  
5         adhesive force of said seta.

1 7. The method of claim 6 wherein said adhesive force is greater than the  
2 cumulative force of said applying and pulling steps.

1 8. A method of fabricating an adhesive microstructure, said method comprising  
2 the steps of:  
3 fabricating an array of shafts; and  
4 forming spatulae on said array of shafts.

1 9. The method of claim 8 wherein said fabricating step includes the step of  
2 constructing said array of shafts with a sandwich of nitride and oxide.

1 10. The method of claim 8 wherein said fabricating step includes the step of  
2 constructing said array of shafts with a sandwich of polymer layers.

1 11. The method of claim 10 wherein said constructing step includes the step of  
2 constructing said array of shafts with a sandwich of polymer layers, wherein a  
3 polymer layer is a spin-cast polymer material selected from the group consisting of  
4 photoresist, polyimide, glass, and epoxy-based compounds.

1 12. The method of claim 10 wherein said constructing step includes the step of  
2 constructing said array of shafts with a sandwich of polymer layers, wherein a  
3 polymer layer is a spray-deposited polymer material selected from the group  
4 consisting of photoresist, polyimide, glass, and epoxy-based compounds.

1 13. The method of claim 10 wherein said constructing step includes the step of  
2 constructing said array of shafts with a sandwich of polymer layers including ultra  
3 violet curable epoxies.

1 14. A fabricated microstructure, comprising:  
2 a shaft with a length of less than 500 microns, said shaft having a diameter of  
3 between 0.01 and 0.1 times said length of said shaft; and

4           an array of spatulae formed at an end of said shaft, said array of spatulae  
5   having a width of less than 10 microns, individual spatula of said spatulae including a  
6   curved segment of a sphere at a spatula terminal end.

1   15.   The fabricated microstructure of claim 14 wherein said shaft has a length of  
2   between approximately 10 and 100 microns.

1   16.   The fabricated microstructure of claim 14 wherein said shaft has a diameter of  
2   approximately 0.05 times said length of said shaft.

1   17.   The fabricated microstructure of claim 14 wherein said curved segment of a  
2   sphere at a spatula terminal end has a radius of approximately 2 $\mu$ m.

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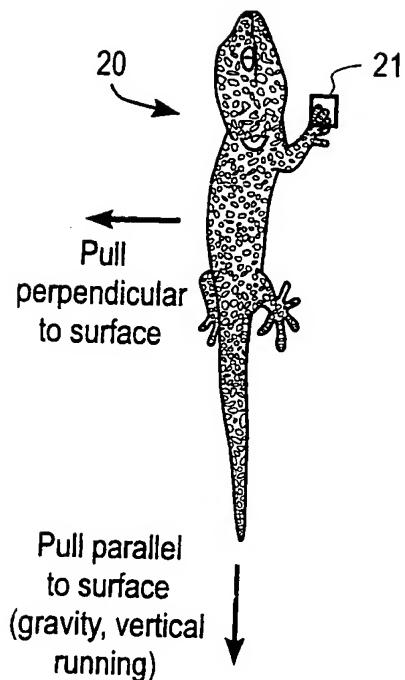


FIG. 1A

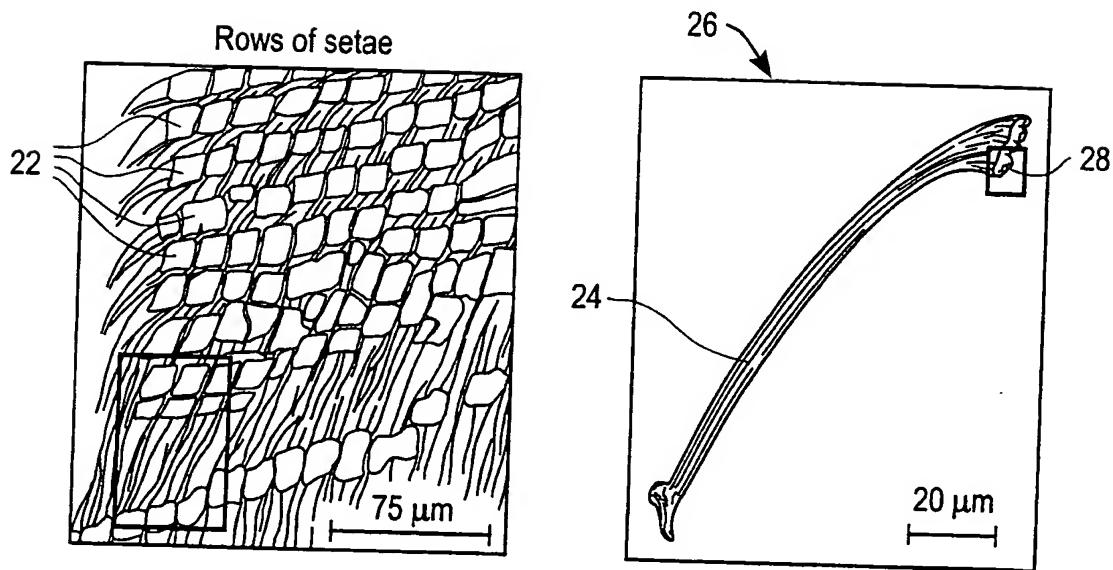


FIG. 1B

FIG. 1C

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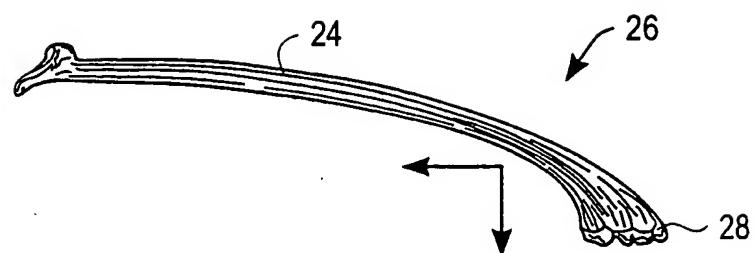


FIG. 1D

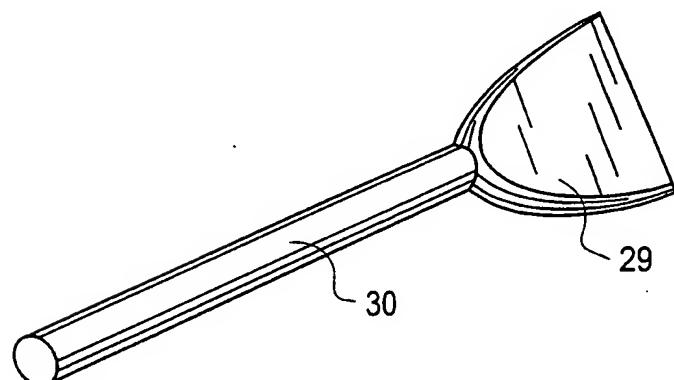


FIG. 1E

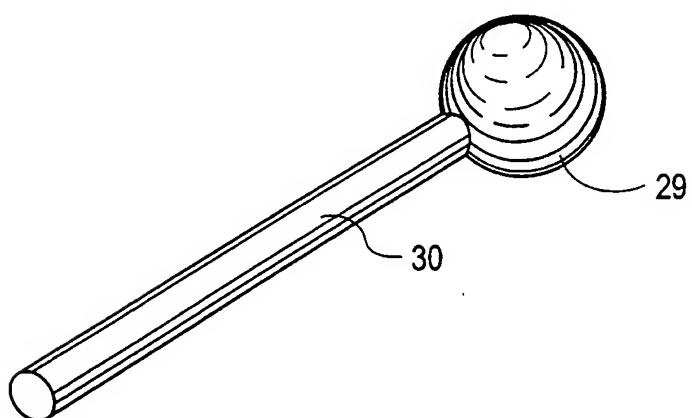


FIG. 1F

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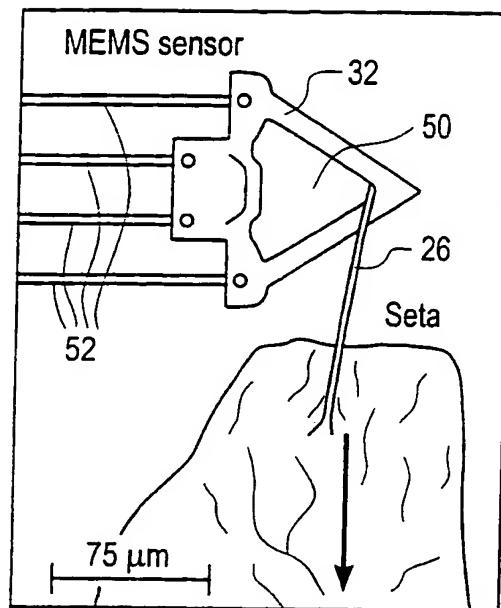
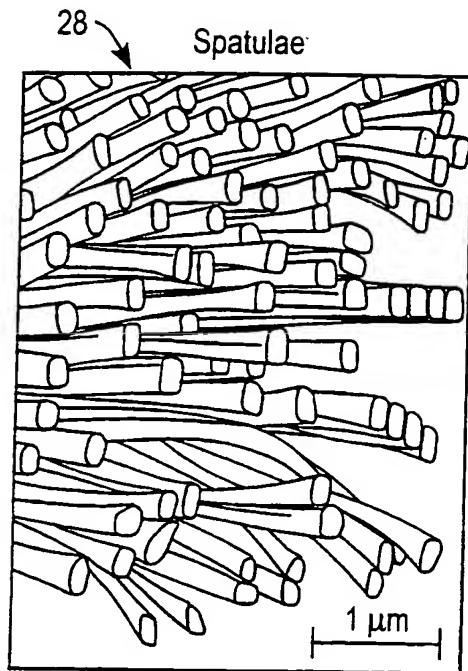


FIG. 1G

FIG. 1H

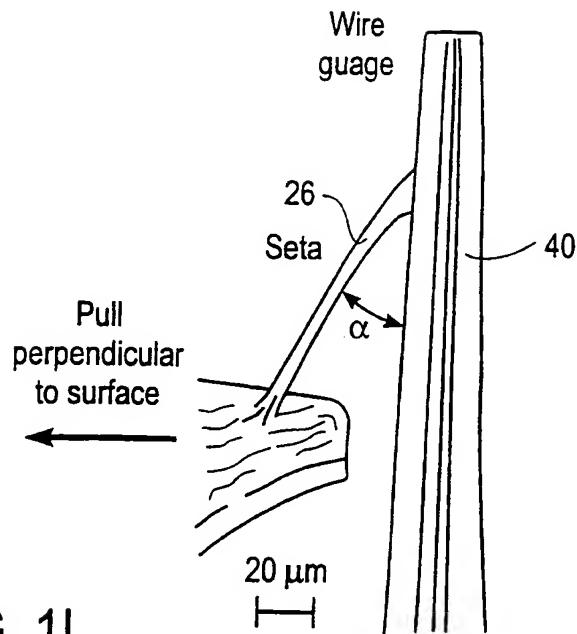


FIG. 1I

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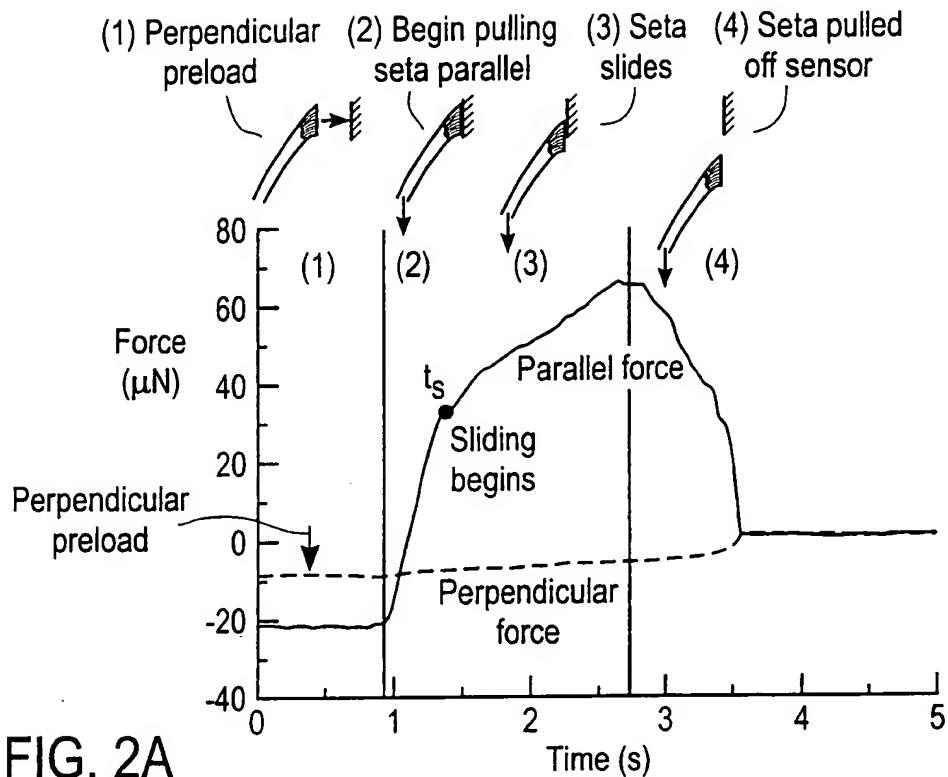


FIG. 2A

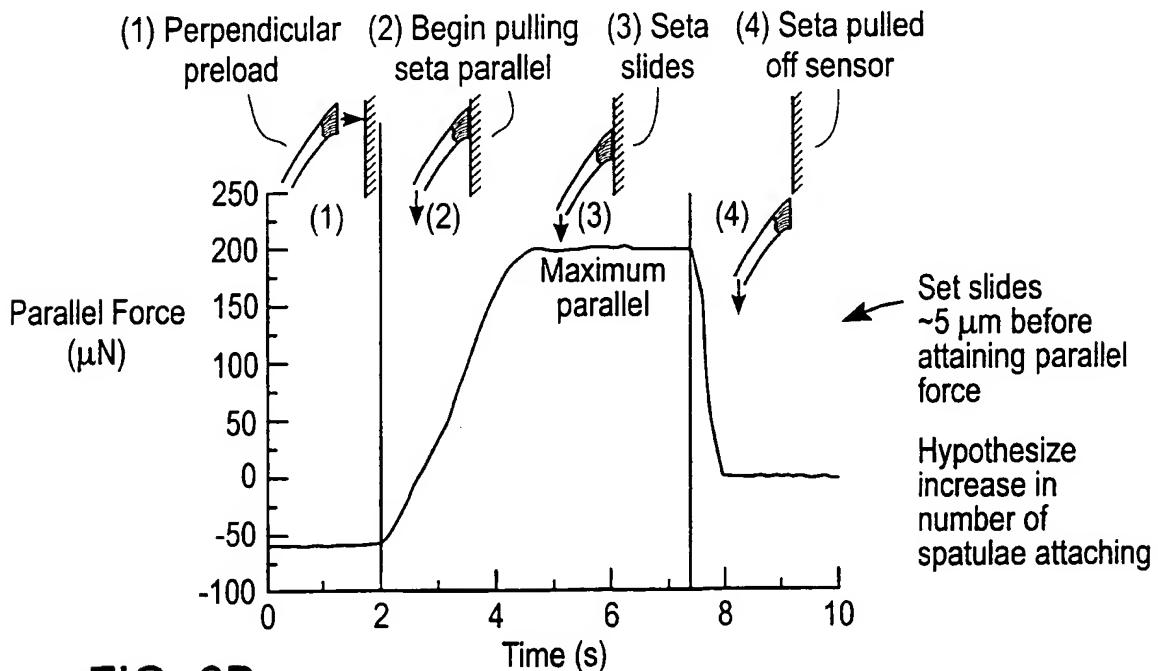


FIG. 2B

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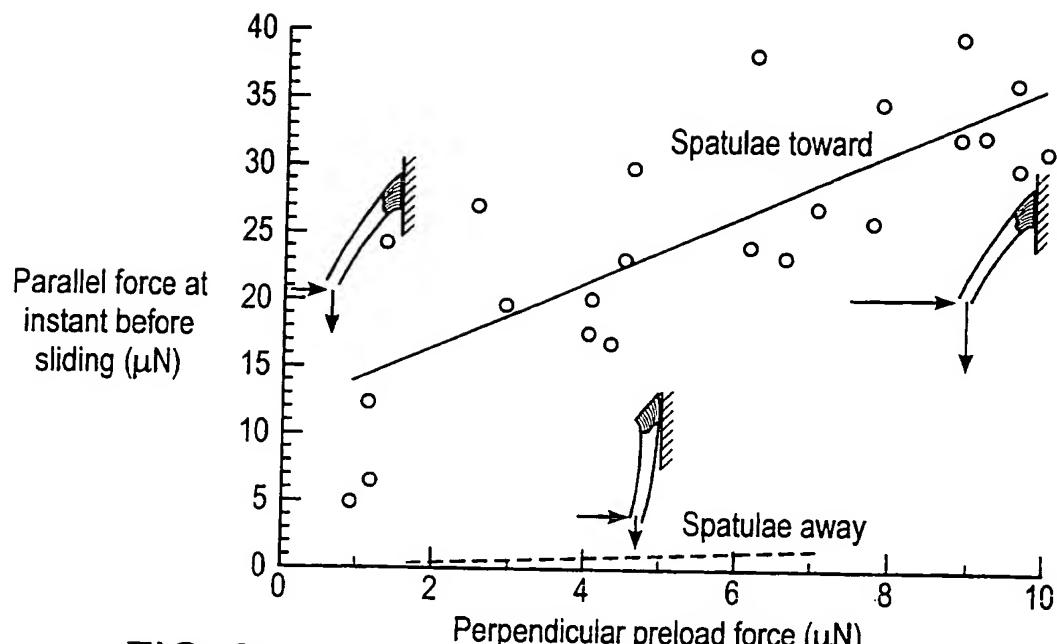


FIG. 3

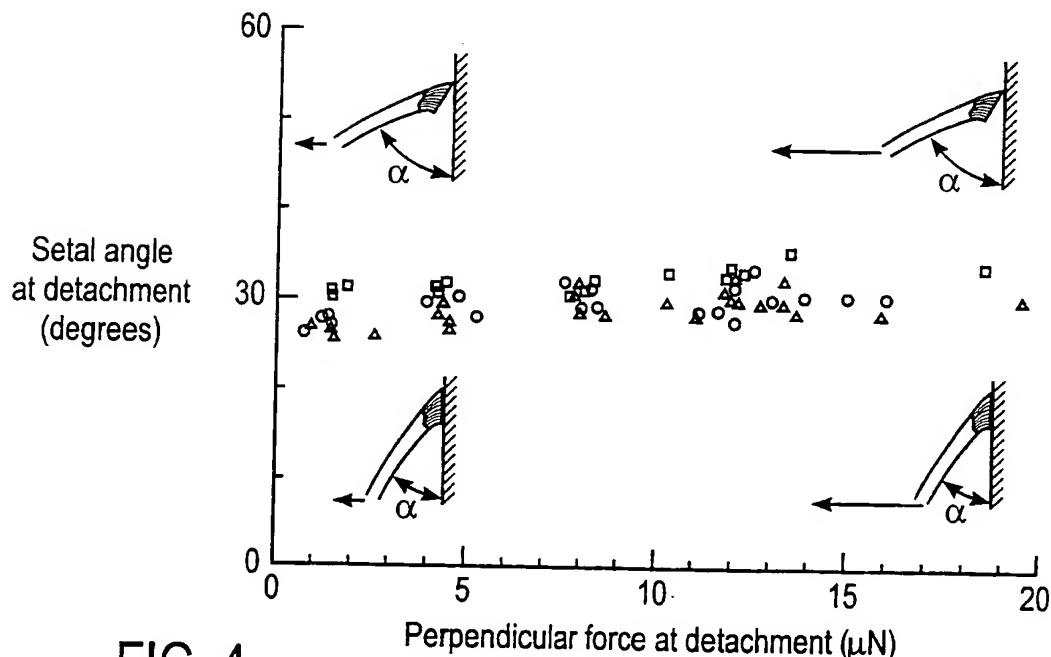


FIG. 4

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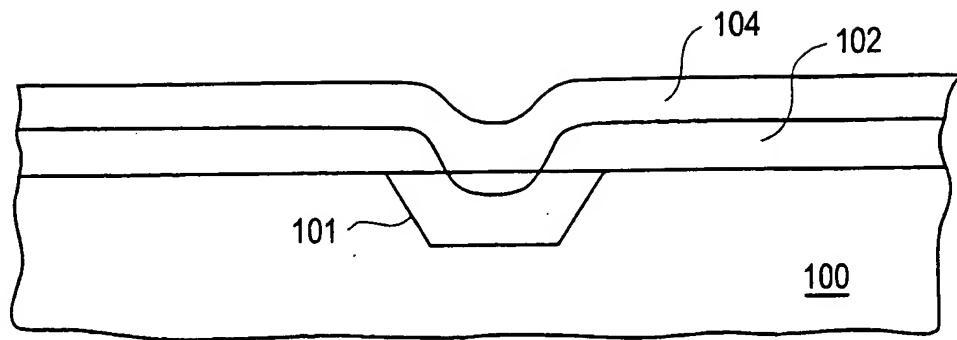


FIG. 5A

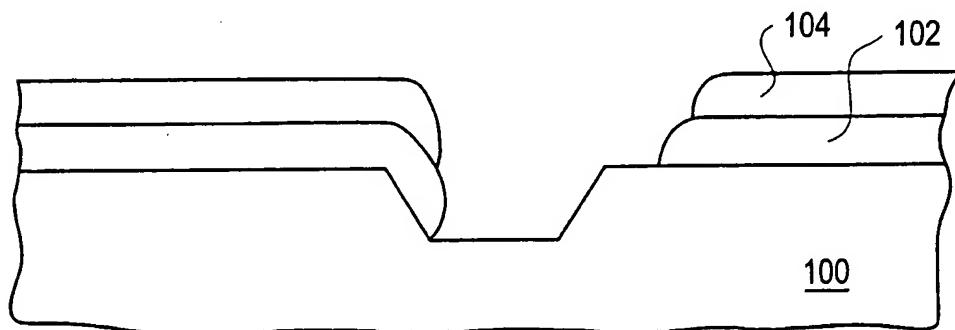


FIG. 5B

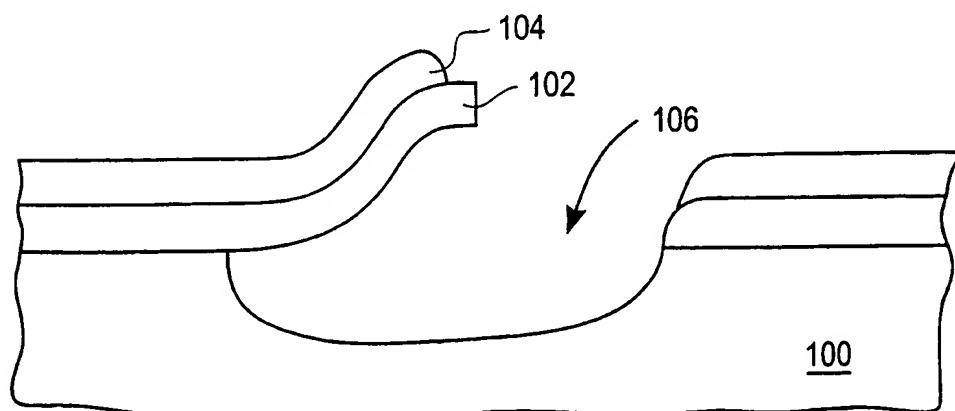


FIG. 5C

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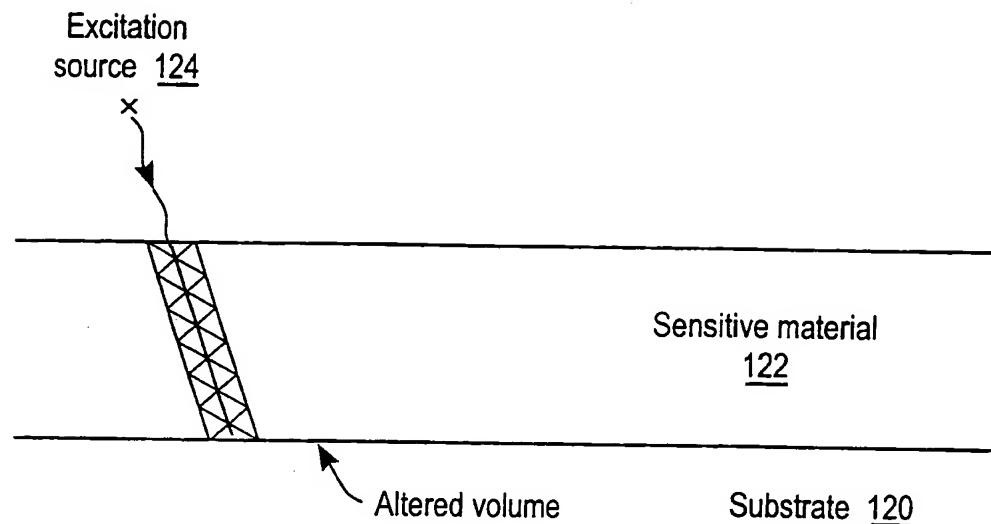


FIG. 6A

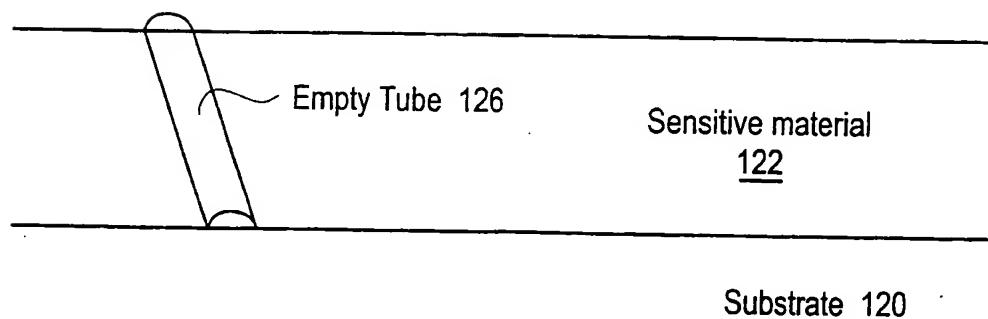


FIG. 6B

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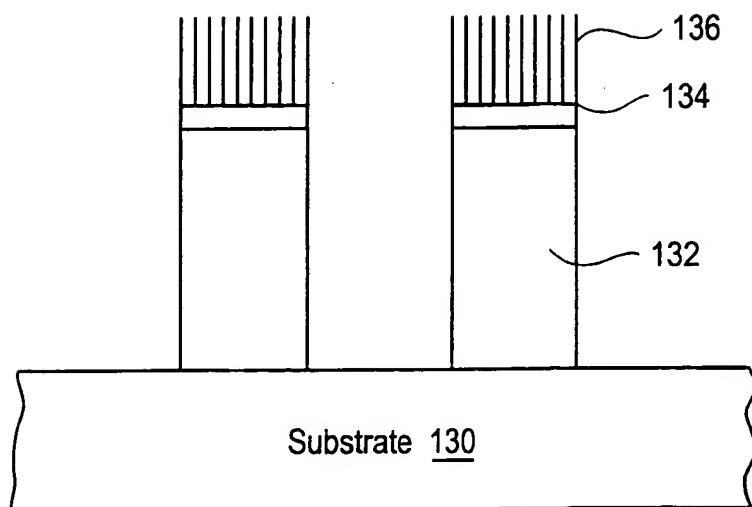


FIG. 7

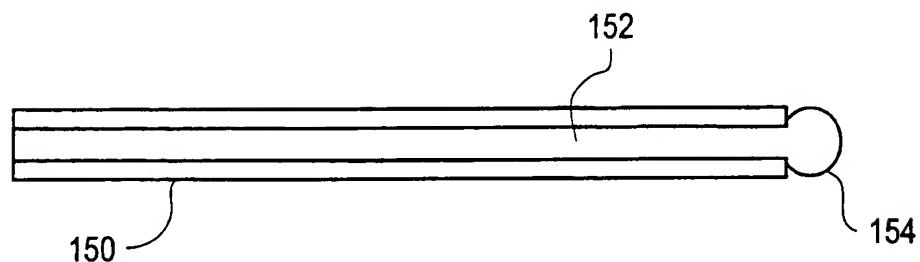


FIG. 8

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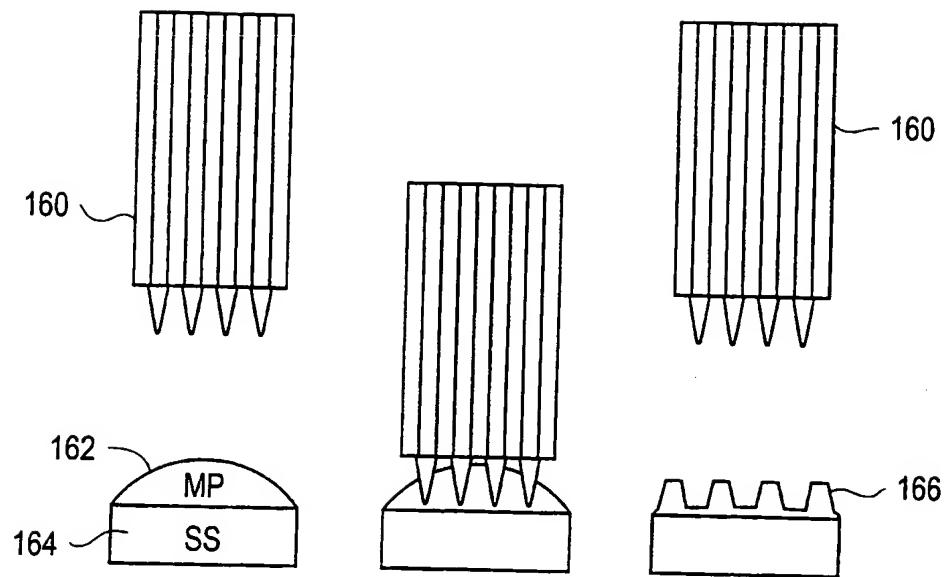


FIG. 9

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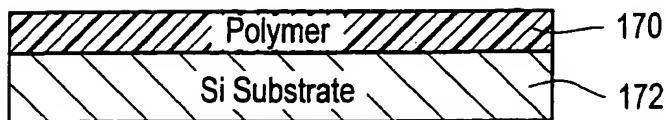


FIG. 10A

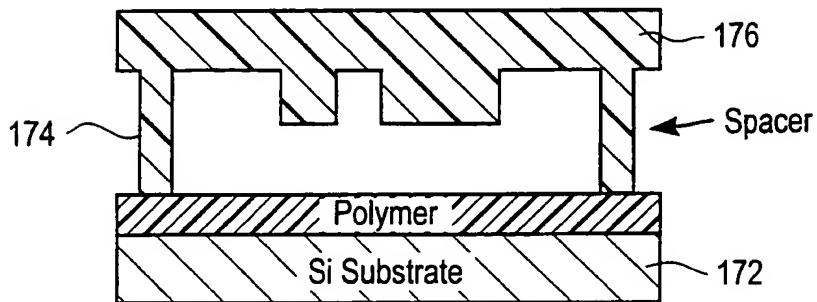


FIG. 10B

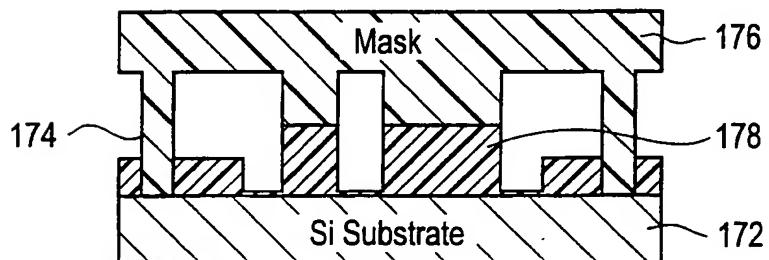


FIG. 10C

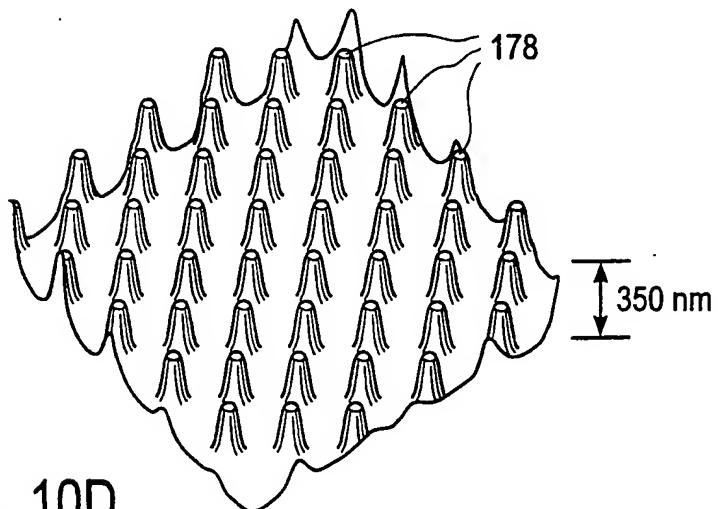


FIG. 10D

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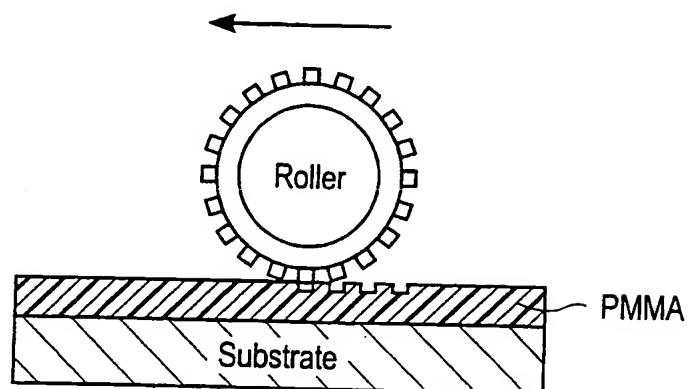


FIG. 11A

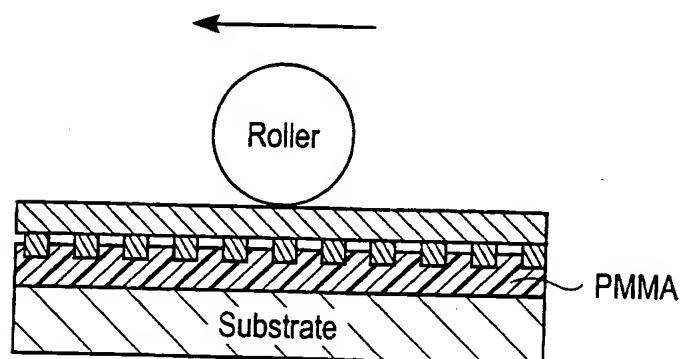


FIG. 11B

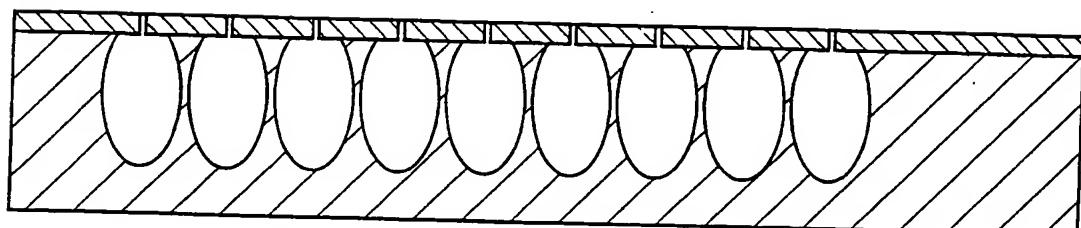


FIG. 12

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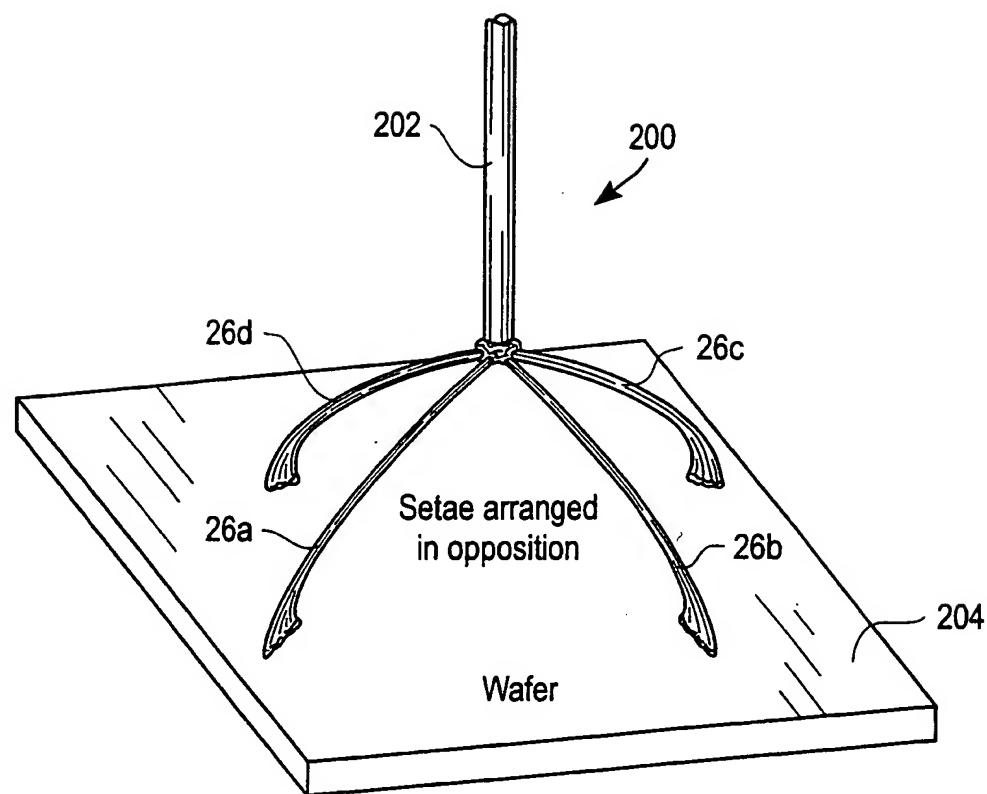
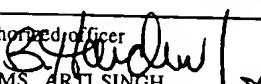


FIG. 13

# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US00/33495

<b>A. CLASSIFICATION OF SUBJECT MATTER</b>																							
IPC(7) :Please See Extra Sheet. US CL :Please See Extra Sheet.																							
According to International Patent Classification (IPC) or to both national classification and IPC																							
<b>B. FIELDS SEARCHED</b>																							
Minimum documentation searched (classification system followed by classification symbols)																							
U.S. : Please See Extra Sheet.																							
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched <b>PALM SERACH BY INVENTOR</b>																							
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) <b>EAST</b>																							
<b>C. DOCUMENTS CONSIDERED TO BE RELEVANT</b>																							
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.																					
A	US 4,545,831A (ORNSTEIN) 08 October 1985. see entire document.	1-17N																					
A	US 5,843,657A (LIOTTA et al.) 01 December 1998, see entire document.	1-17																					
<input type="checkbox"/> Further documents are listed in the continuation of Box C. <input type="checkbox"/> See patent family annex.																							
<table border="0" style="width: 100%; border-collapse: collapse;"> <tr> <td style="width: 15%;">* Special categories of cited documents:</td> <td style="width: 15%;">*T*</td> <td style="width: 70%;">later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</td> </tr> <tr> <td>*A*</td> <td></td> <td>document defining the general state of the art which is not considered to be of particular relevance</td> </tr> <tr> <td>*E*</td> <td>*X*</td> <td>earlier document published on or after the international filing date</td> </tr> <tr> <td>*L*</td> <td></td> <td>document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</td> </tr> <tr> <td>*O*</td> <td>*Y*</td> <td>document referring to an oral disclosure, use, exhibition or other means</td> </tr> <tr> <td>*P*</td> <td></td> <td>document published prior to the international filing date but later than the priority date claimed</td> </tr> <tr> <td></td> <td>*&amp;*</td> <td>document member of the same patent family</td> </tr> </table>			* Special categories of cited documents:	*T*	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention	*A*		document defining the general state of the art which is not considered to be of particular relevance	*E*	*X*	earlier document published on or after the international filing date	*L*		document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	*O*	*Y*	document referring to an oral disclosure, use, exhibition or other means	*P*		document published prior to the international filing date but later than the priority date claimed		*&*	document member of the same patent family
* Special categories of cited documents:	*T*	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention																					
*A*		document defining the general state of the art which is not considered to be of particular relevance																					
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*P*		document published prior to the international filing date but later than the priority date claimed																					
	*&*	document member of the same patent family																					
Date of the actual completion of the international search  31 MAY 2001	Date of mailing of the international search report  <b>21 JUN 2001</b>																						
Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-3230	Authorized officer  MS. ARTI SINGH Telephone No. 703-305-0661																						

# INTERNATIONAL SEARCH REPORT

International application No.

PCT/US00/33495

## A. CLASSIFICATION OF SUBJECT MATTER:

IPC (7):

A 01 K 1/03;  
A 01 N 1/00, 3/00;  
B 32 B 31/14;  
C 12 N 5/00.

## A. CLASSIFICATION OF SUBJECT MATTER:

US CL :

119/417, 423;  
427/4;  
428/16, 343;  
435/6.1, 325, 1.17;  
156/57, 61, 62.2, 68, 77, 241;  
424/81, 3.

## B. FIELDS SEARCHED

Minimum documentation searched

Classification System: U.S.

119/417, 423;  
427/4;  
428/16, 343;  
435/6.1, 325, 1.17;  
156/57, 61, 62.2, 68, 77, 241;  
424/81, 3.

